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HEADQUARTERS
U.S. ARMY ARMAMENT,
MUNITIONS AND CHEMICAL COMMAND

FINAL REPORT

DEVELOPMENT OF
METHODOLOGY AND TECHNOLOGY
FOR
IDENTIFYING AND QUANTIFYING
EMISSION PRODUCTS
FROM
OPEN BURNING AND OPEN DETONATION
THERMAL TREATMENT METHODS.

BANGBOX TEST SERIES

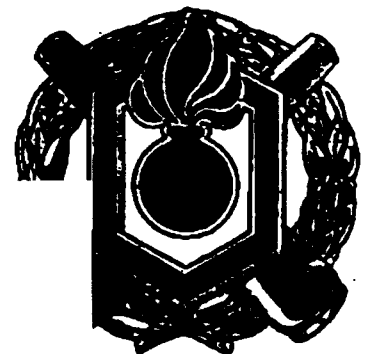
VOLUME 3
QUALITY ASSURANCE AND QUALITY CONTROL

JANUARY 1992

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19. ABSTRACT (Continue on reverse if necessary and identify by block number) The 1989 BangBox test validated technologies and methodologies proposed for identifying and quantifying emissions resulting from the open burning (OB) of propellants and open detonation (OD) of explosives. This test also provided the first credible data on combustion products resulting from OB/OD treatment procedures. The vigorous quality assurance/quality control (QA/QC) program established well before the first trial was continued through the reporting phase. Major elements of the QA/QC program were a quality assurance program plan (QAPP), letters of instruction (LOI) covering all technical aspects of the testing program, a sample identification and tracking system, test monitoring by the scientific support contractor, visits to the test site and assay laboratories by an independent QA contractor, assays by different laboratories using different instruments (gas chromatograph/mass spectrometer and supercritical fluid chromatograph/mass spectrometer), equipment audits by the U.S. Environmental Protection Agency's Atmospheric Research and Exposure Assessment Laboratory (AREAL), assay of samples spiked by the AREAL, and cooperative assistance from the U.S. Army Toxic and Hazardous Material Agency, and the U.S. Army Environmental Hygiene Agency. (Cont'd on reverse).					
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19. (Cont'd)

Conduct and results of this QA program are included in this volume, along with the QAPP, the Test Design Plan, and LOI's which support this test.

QUALITY ASSURANCE STATEMENT
OB/OD BANGBOX REPORT

This report of the Quality Assurance Unit summarizes the review of the BangBox Final report. The portion of the report furnished to ELI for review consisted of the Test Summary (Volume 1); Test Development (Volume 2); and Quality Assurance/Quality Control (Volume 3).

The information contained in these sections of the report was reviewed for completeness and accuracy. This was accomplished by verification of the information contained in the report against the records of data collected during the testing phase. As it was neither realistic nor feasible to review all information contained in the final report, randomly selected material was reviewed. This consisted of approximately ten percent of all material furnished.

No appreciable discrepancies were found during this review process. We therefore feel that the final report as furnished to us for review represents an accurate and complete reporting of the information obtained during the OB/OD BangBox tests.

ORIGINAL SIGNED

Floyd W. McMullin Jr.
Quality Assurance Officer

Date _____

ORIGINAL SIGNED

Gary M. Booth
Quality Assurance Director

Date _____

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FOREWORD

A test planning directive to conduct the OB/OD test in support of U.S. Army Armament, Munitions and Chemical Command (AMCCOM) was issued by U.S. Army Test and Evaluation Command (TECOM) on 28 April 1988¹. This test was conducted following the Technical Steering Committee Symposium which was convened in July 1988. The requirement for identifying and quantifying emissions from the open detonation of explosives and open burning of propellants was discussed in detail by authorities from throughout the military, academic, and commercial communities. Conclusions and recommendations developed during the symposium are reported in proceedings of the symposium².

The BangBox Test was the basis for the selection of collection and analysis methods for large-scale field tests.

The BangBox Test series report includes three volumes:

Volume 1. A summary which describes the planning phase, the conduct of trials, sample analyses and results, and the conclusions and recommendations. It is useful for those who need the background, synopsized results, conclusions, and recommendations without the complete details with the supporting data and information.

Volume 2. A stand-alone document which covers the detail of the complete test. It describes the test development, description of the test materiel, and the trial results as they relate to the test objectives and the explosives and propellants tested.

¹Letter, AMSTE-TA-F, Headquarters, U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland, 20 April 1988, subject: Test Planning Directive for Special Study of Open Burning/Open Detonation (OB/OD). Phase II, TECOM Project No 2-CO-210-000-017.

²*Proceedings of the Technical Steering Committee Symposium 6-8 July 1988*, Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

Volume 3. The quality assurance/quality control (QA/QC) report covers the quality assurance project plan, the test design plan, the letters of instruction (prepared for procedural instruction), the quality audits, the reports of the quality audits, and the results of the blind spikes analyzed by the laboratories.

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SECTION 1. INTRODUCTION

1.1 Open burning and open detonation (OB/OD) is currently the primary means of demilitarization employed by the Department of Defense (DoD) for the treatment of explosive residue, propellants, and munitions.

1.2 The increasing need for data on OB/OD combustion products to support environmental documentation requirements, such as those of the Clean Air Act (CAA), Clean Water Act (CWA), and Resource Conservation and Recovery Act (RCRA) resulted in a critical need for a test program to collect data to be used as a basis for informed decisions concerning the limitations and restrictions of OB/OD, the need for alternative methods where required, and maintaining an effective, economical, and environmentally safe means of accomplishing the required demilitarization/treatment. The Single Manager for Conventional Ammunition within the DoD began to address this need in the early 1980's, and to provide scientific data to answer the question of environmental acceptability of OB/OD thermal treatment methods.

1.3 Because of the scope of the OB/OD project, the effort was divided into several Phases. The initial effort included outdoor OB/OD operations at Tooele Army Depot involving a wide variety of explosives, propellants and munitions. After evaluating the data generated from this work and considering the lessons learned, a large, highly qualified group (the Technical Steering Committee (TSC)) was assembled to plan follow-on phases.

1.4 Although limited data are available from past studies on the generation of particulates and criteria pollutants from small-scale laboratory and field OB/OD operations, little field data are available on the levels of semivolatile organic emissions that result from unconstrained combustion of propellants by open-air burning or of explosives by open-air detonation. The semivolatile compounds are very difficult to collect and identify. Thus, prior to the conduct of full-scale OB/OD field testing operations involving large quantities (thousands of kilograms) of explosives and propellants, a limited number of small-scale explosive detonation and burning trials was conducted within a controlled, ambient air environment. The test chamber that was used to provide this controlled environment was operated by Sandia National Laboratories (SNL), Kirtland Air Force Base (AFB), New Mexico, and is locally known as the BangBox (BB).

1.5 The BB is an air-supported hemisphere of coated fabric approximately 950 m³ in volume that can be highly instrumented during detonations of half pound-sized explosives or pound-size propellant burns.

1.6 The purpose of testing in the BB was to confine OB and OD emissions to enable sampling over a long enough period that the concentration of the emittants in the samples would be adequate for the detection and quantification of trace levels of organic compounds, metals, soot, and criteria pollutants. The BB trials incorporated a redundancy in sampling and analytical techniques to allow the comparison of alternative sampling and analysis equipment and methods as well as the evaluation of the accuracy and precision of sampling equipment to be used later in an instrumented Fixed Wing Aircraft (FWAC) on large-scale outdoor OB/OD trials.

1.7 Three familiarization trials were run in the BB during 1, 5, and 7 December 1988. Two of these trials involved detonation of 0.5 lb 2,4,6-trinitrotoluene (TNT) simultaneously with a release of SF₆ tracer gas. The third involved release of SF₆. The results of these trials allowed a preliminary characterization of chamber homogeneity, volume, and ventilation rate, and the final selection of sampling equipment and analysis procedures to be used on remaining OB/OD tests.

1.8 The main series of eight data collection OB/OD trials in the BB was accomplished 31 January to 16 February 1989 with 0.5 lb blocks of TNT or 1 lb each of a double-base and a composite propellant. TNT was the explosive of choice since it is the most oxygen-deficient of the military explosives (minus 78 percent by weight). It was most likely to produce significant amounts of the products of incomplete combustion. Propellants selected included a double-base formulation containing primarily nitrocellulose, nitroglycerin, and ethyl cellulose, and a composite propellant containing large amounts of ammonium perchlorate.

1.9 Product compositions and yields could be compared with results reported from earlier laboratory-scale testing, less controlled open-air detonations, and from computer-modeled combustion product studies.

1.10 Future work (June 1989) will include large-scale field test series using 2000-lb explosive detonations and 7000-lb propellant burns at Dugway Proving Ground (DPG) in Utah.

1.10.1 The purpose of the field test series A will be to verify BB conclusions by sampling real-world OB- and OD-generated combustion product clouds with an instrumented Fixed Wing Aircraft (FWAC) developed by the Atmospheric Research Group at SNL. (This aircraft has been employed on numerous previous air pollution studies.)

1.10.2 Phase B at DPG will expand Phase A work to include ejecta and fallout soil sampling.

1.10.3 Phase C, scheduled to begin late-summer 1990, will comprise the main data collection effort on outdoor, large-scale OB/OD operations encompassed by this phase of the project.

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SECTION 2. PROJECT DESCRIPTION

2.1 Purpose Of Investigation

The OB/OD BB test series was designed to develop, verify and confirm the OB/OD thermal treatment method test technology/methodology.

2.1.1 Objectives

2.1.1.1 Characterize the BB chamber volume, ventilation rate, and combustion product cloud homogeneity level.

2.1.1.2 Develop and improve proposed air sampling equipment and sample analysis procedures to be used in later phases on the FWAC, for sampling product clouds from large-scale follow-on field OB/OD trials.

2.1.1.3 Refine, standardize, and compare supercritical-fluid chromatography (SFC) and gas chromatography (GC) techniques for extracting and analyzing resins, filters, and soils for trace quantities of semivolatile organic OB/OD combustion products and residues, using mass spectrometer (MS) detectors.

2.1.1.4 Verify adequacy of other standard analytical methods to be used for analyses of gases, particulates, volatile organic compounds, metals, and nonmetals.

2.1.1.5 Identify and quantify specific target analytes for TNT, a double-base propellant, and a composite propellant.

2.1.1.6 Assess polychlorinated dibenzodioxins (PCDD) and dibenzofurans (PCDF) levels generated from burning the composite propellant containing high concentration of NH_4ClO_4 .

2.1.1.7 Provide information on the morphology, composition, and size distributions of airborne particulate material generated by OB/OD operations in the BB.

2.1.1.8 Examine, using data produced under controlled conditions, the validity of the proposed Carbon Balance method of calculating emission factors; compare the results with those calculated using the more-conventional cloud volume times concentration method.

2.1.1.9 Identify or develop appropriate program-specific QA/QC procedures.

2.1.1.10 Develop and establish procedures for transport and storage of sample specimens. The OB/OD trials conducted in the BB were designed to attain the following Objectives:

2.2 Project Planning

2.2.1 The BB trials were planned by the TSC whose expertise covered a wide range of pertinent disciplines, including sampling technology, chemical analysis, organic and inorganic chemistry, explosives, environmental chemistry and regulations, and statistical analysis. This spectrum of capabilities ensured that the experimental approach would be appropriate for the objectives.

2.2.2 The TSC continued to guide the progress of the investigation and to recommend refinements in experimental details during the course of the BB trials.

2.2.3 Nationally-recognized authorities and laboratories were contracted to participate in the test execution and sampling, and to perform the required sample analyses.

2.2.4 Personnel from EPA's Quality Assurance Division of the Atmospheric Research and Exposure Assessment Laboratory of EPA at Research Triangle Park, NC, (EPA-QAD) provided advice and audit services.

2.2.5 The quality of the planning, guidance, and working-level personnel laid a solid foundation for assuring high quality data from the BB investigation.

2.2.6 The basic BB trial series planning document is the AMCCOM Test Design Plan (Appendix A of this volume).

2.3 Project Execution

2.3.1 Chronology of BB Trials

2.3.1.1 Table 2.1 outlines the sequence of trials conducted in the BB. Volume 2 of the AMCCOM BB report describes each trial in detail (starting on page 2-1).

2.3.1.2 Tables 2.2a and 2.2b show a typical sequence of events for the TNT detonation trials. The BB chamber was cleaned before the trials by washing the interior and vacuuming the floor; it was also vacuumed between each detonation trial and washed between differing kinds of tests.

Table 2.1. BangBox Trial Schedule and Synopsis.

Date	Activity	Primary Goals
1 Dec 88	0.5 lb TNT OD, SF ₆ release	Chamber volume and cloud homogeneity determination.
5 Dec 88	SF ₆ release	Chamber ventilation rate determination.
7 Dec 88	0.5 lb TNT OD, SF ₆ release	Check out setup, equipment, procedures.
31 Jan 89	0.5 lb TNT OD, SF ₆ release	Analyte concentration; chamber air homogeneity, volume, & ventilation rate.
2 Feb 89	0.5 lb TNT OD, SF ₆ release	Analyte concentration; chamber air homogeneity, volume, & ventilation rate.
6 Feb 89	0.5 lb TNT OD, SF ₆ release	Analyte concentration; chamber air homogeneity, volume, & ventilation rate.
7 Feb 89	8-h Background air sample	Analyte concentration inside and outside BB.
8 Feb 89	8 Consecutive 0.5 lb TNT ODs	Analytes from all detonations collected on same samplers (to increase sample concentrations).
9 Feb 89	1.0 lb Double-Base Propellant Burn, SF ₆ release	Analyte concentration.
13 Feb 89	0.5 lb TNT, Aqueous Foam-Mitigated OD	Analyte concentrations under attenuated blast conditions.
15 Feb 89	0.5 lb TNT OD, SF ₆ release	Analyte Concentrations from simultaneous collection in 27 32-L tanks ("Big Gulp" trial).
16 Feb 89	1.0 lb Composite Propellant Burn, SF ₆ release	Analyte concentration and special sampling for selected polychlorinated dibenzodioxins and dibenzofurans.

Table 2.2a. Typical Chronology of Events for a TNT Detonation Trial.

Timing (min)	Activity
t-75	Install background filters, semi-VOST cartridges, bubblers. Measure and record initial flow rates on all samplers. Check zero and span on all continuous monitors.
t-60	Clear all personnel from chamber interior. Begin background sampling with all instruments and samplers. Start standard video.
t-30	Complete background sampling. Record final flow rates on all samplers. Remove background sampling media. Install all test filters, semi-VOST cartridges, bubblers, etc. Record initial flow rates on all samplers.
t-15	Hang 0.5 lb TNT charge in test fixture.
t-5	Start data acquisition on all continuous instruments. Clear all personnel to safe building. Connect detonator cable to charge and arm.
t=0	Detonate charge. Run high speed camera (Jan 7 only).
t+3	Remotely start all filter and semi-VOST samplers inside chamber. Collect nonhomogeneous sample No. 1 in sampling bag. Pump bag sample through sampling media in airlock. Switch criteria gas monitors to sample directly from bag for 3 min. Fill 6-L evacuated cylinder from interior of chamber. Fill 6-L evacuated cylinder from bag.
t+5	Switch mixing fans on for 3 min.
t+10	Change sampling media on bag system.
t+15	Collect homogeneous (mixed) air sample No. 1 in sampling bag. Pump bag sample through sampling media. Switch criteria gas monitors to sample directly from bag for 3 min. Fill 6-L evacuated cylinder from interior of chamber. Fill 6-L evacuated cylinder from bag.
t+25	Change sampling media on bag system.

Table 2.2b. Typical Chronology of Events for a TNT Detonation Trial.

Timing (min)	Activity
t+30	Collect mixed sample No. 2 in sampling bag. Pump bag sample through sampling media. Switch criteria gas monitors to sample directly from bag for 3 min. Fill 6-L evacuated cylinder from chamber interior. Fill 6-L evacuated cylinder from bag.
t+35	Stop direct semi-VOST, filter, and bubbler samplers. Stop video. Re-enter chamber and measure final sampler flows.
t+45	Check zero and span on all continuous monitors. Retrieve and backup all test data. Collect and preserve all sampling media.

2.3.1.3 Samples from the trials were collected, sealed, and transported to the appropriate chemical laboratories for extraction, concentration, and analysis, according to the schedule delineated in Tables 2.3a and 2.3b.

2.3.1.4 Alpine West Laboratories (AWL), Provo, UT, cleaned the XAD-2™ resin used in the semi-VOST samplers, and performed SFC-MS analyses of semivolatile organic compounds in assigned semi-VOST resin and quartz filter extracts.

2.3.1.5 Battelle-Columbus Division (BCD), Columbus, OH, analyzed semi-VOST resin and quartz-fiber filter extracts for target semivolatile organic compounds using GC-MS analyses. Personnel from BCD also collected other samples and analyzed them for PCDDs and for PCDFs.

2.3.1.6 Lawrence-Berkeley Laboratory (LBL), Berkeley, CA, used x-ray fluorescence analysis to measure the quantities of metals and selected non-metals on Teflon™ filter samples.

Table 2.3a. Typical Chronology for BB Trial Samples.

Sample Type	Destination	Shipped	Method	Received	Extracted	Analyzed	Comments
Quartz-Fiber Filter	AWL	t+5 d	Courier or express carrier.	24 to 48 h after shipment.	Within 7 days of receipt.	As lab schedule permitted; was up to 60 days following extraction.	Some extracts split and forwarded to BCD for analysis.
	BCD	t+5 d	Courier or express carrier.	24 to 48 h after shipment.	Within 14 days of receipt.	As lab schedule permitted; was up to 45 days following extraction.	
	SSL	Within 7 d of receipt at AWL.	Express carrier.	24 to 48 h after shipment.	NA*	Within 72 h of receipt.	
XAD-2 Resin	AWL	t+5 d	Courier or express carrier.	24 to 48 h after shipment.	As schedule permitted; was up to 28 days.	As lab schedule permitted; was up to 60 days following extraction.	
	BCD	t+5 d	Courier or express carrier.	24 to 48 h after shipment.	As schedule permitted; was up to 28 days.	As lab schedule permitted; was up to 45 days following extraction.	
Polyurethane Foam Filter (PUFF)	BCD	t+1 d	Courier.	<8 h after shipment.	Unknown	Unknown	
	LBL	t+30 d	Express carrier.	24 to 48 h after shipment.	NA	<72 h after receipt.	
Teflon™							

Table 2.3b. Typical Chronology for BB Trial Samples.

Sample Type	Destination	Shipped	Method	Received	Extracted	Analyzed	Comments
Polycarbonate Filters	SNL Scanning Electron Microscope Lab	t + 2 h	Courier	< 1 h after shipment.	NA	As lab schedule permitted; was up to 21 days from receipt.	
Glass Impingers (Bubblers)	SNL Industrial Hygiene Lab	t + 2 h	Courier	< 1 h after shipment.	NA	< 12 h after receipt.	
0.85-L SS SF ₆	OGC	NA	NA	NA	NA	< t + 24 h	Analysis on site at SNL BB facility.
6-L SS VOC	OGC	t + 1 d	Express carrier.	24 to 48 h after shipment.	NA	As lab schedule permitted; was up to 14 days after receipt.	
32-L SS	OGC	t + 1 d	Express carrier.	24 to 48 h after shipment.	As processing of tanks permitted; was up to 28 days from receipt.	NA	Extracts forwarded to AWL & BCD for analysis.

*Not applicable.

2.3.1.7 Oregon Graduate Center (OGC), Beaverton, OR, prepared and operated the evacuated SS canisters samplers at the BB, and analyzed their contents for volatile organic compounds. OGC personnel also released and analyzed SF₆ to determine chamber volume, mixing rate, and the ventilation rate of the BB.

2.3.1.8 Sunset Laboratories (SSL), Forest Grove, OR, analyzed an aliquot of selected quartz-fiber filters to determine the quantities of elemental, organic, and inorganic carbon.

2.3.1.9 Sandia National Laboratories (SNL), Albuquerque, NM, operated the BB, ran the real-time instruments to measure the level of criteria pollutants and particulates, and operated and analyzed bubbler samplers to determine certain inorganic gases (NH₃, HCN, and HCl).

2.3.2 Chronology of QA Activities

ELI performed on-site compliance audits and accomplished other QA activities on the dates listed in Tables 2.4a, 2.4b, and 2.4c. The EPA-QAD performance audit of SNL is included. Section 3 presents the findings from the audits.

Table 2.4a. Abstract of QA Activities.

Date	Location	Person (ELI staff)	Purpose
17 Nov 88	Dugway Proving Ground, UT	Todd Parrish	Accompany representatives of DPG and AWL to obtain soil samples for EPA spiking and for use as soil blanks in laboratory.
30 Nov 88	Sandia National Lab	Gary Booth Todd Parrish Floyd McMullin	Pre-test meeting of QA personnel. Todd Parrish observed the CO ₂ sampling in the air building (BangBox).
1 Dec 88	Sandia National Lab	Gary Booth Todd Parrish Floyd McMullin	Preliminary meeting of all personnel. Preliminary test detonation. Sample collection observed by QA personnel.
2 Dec 88	Sandia National Lab	Gary Booth Todd Parrish Floyd McMullin	Todd Parrish and Floyd McMullin conducted logbook audit of SNL instrumentation located in the BangBox. General meeting of all personnel in follow-up to preliminary detonation of 1 Dec 88.
3 Dec 88	Sandia National Lab	Gary Booth Todd Parrish Floyd McMullin	General meeting of test personnel to discuss QA project plan and test design plan.
5 Dec 88	Sandia National Lab	Todd Parrish Floyd McMullin	Met with OGC representative. Completed documentation of samples from preliminary test. Reviewed sample numbering assignments and status of LOI from various facilities.
6 Dec 88	Sandia National Lab	Todd Parrish Floyd McMullin	General meeting of all participants. Extensive discussion relating to test time table and check-list.
7 Dec 88	Sandia National Lab	Todd Parrish Floyd McMullin	BangBox test preparation observed. Following detonation, samples checked against documentation. Posttest meeting of all personnel held.

Table 2.4b. Abstract of QA Activities.

Date	Location	Person (ELI staff)	Purpose
16 Dec 88	Sandia National Lab	Floyd McMullin	QA site visit with Wayne Einfeld.
28 Dec 88	Battelle Columbus Div.	Todd Parrish	QA site visit with Larry Slivon.
18 Jan 89	Brigham Young University	Todd Parrish	QA audit of Data Management Center files.
20 Jan 89	Brigham Young University	Todd Parrish	QA audit of Statistic Center files.
23 Jan 89	Sunset Laboratory	Floyd McMullin	QA site visit with Robert Cary.
24 Jan 89	Oregon Graduate Center	Floyd McMullin	QA site visit with Robert Dalluge and Rei Rasmussen.
25 Jan 89	Alpine West Laboratory	Todd Parrish	QA site visit with Christine Rouse.
30 Jan 89	Sandia National Laboratory	Gary Booth Todd Parrish	General meeting of test personnel. Observed equipment preparations for test.
31 Jan 89	Lawrence Berkely Labs	Floyd McMullin	QA site visit with Robert Giaque and Linda Sindelar.
31 Jan 89	Sandia National Lab	Todd Parrish	Observed background testing and test detonation. Following test obtained sample documentation.
1 Feb 89	Sandia National Lab	Todd Parrish	Observed equipment preparation for next scheduled test.
2 Feb 89	Sandia National Lab	Todd Parrish	Observed equipment checks and calibrations. Also checked instrument logbooks. Following detonation today observed posttest sample handling.
6 Feb 89	Sandia National Lab	Gary Booth Floyd McMullin	General meeting of all test personnel and members of EPA audit team. Observed preparation of BangBox for detonation. Following test, sample documentation and samples correlated.

Table 2.4c. Abstract of QA Activities.

Date	Location	Person (ELI staff)	Purpose
7 Feb 89	Sandia National Lab	Gary Booth Floyd McMullin	Posttest meeting of all personnel. Long-duration background testing monitored by Floyd McMullin. EPA team performed audit on SNL real time gas instrumentation.
8 Feb 89	Sandia National Lab	Floyd McMullin	General meeting of all test personnel. EPA audit team gave exit briefing. Observed multiple detonation tests.
9 Feb 89	Sandia National Lab	Floyd McMullin	General meeting to plan the double base burn scheduled for today. Observed preparation for and execution of test.
15 Feb 89	Sandia National Lab	Floyd McMullin	Preliminary preparation of documentation of sampling for upcoming tests. Reviewed sample documentation from foam test conducted on 13 Feb 89. Met with BCD personnel on site for upcoming double base propellant burn. Samples and documentation from the Big Gulp test detonation gathered and correlated.
16 Feb 89	Sandia National Lab	Floyd McMullin	Observed preparation for and execution of double base propellant burn. After test samples and documents were collected and correlated.
9 May 89	Sunset Laboratory	Floyd McMullin	QA site visit with Robert Cary.

SECTION 3. EVALUATION OF OPERATIONAL QUALITY

3.1 Documentation and Data Management

3.1.1 Sample Tracking

3.1.1.1 ELI implemented a "chain-of-custody" sample-tracking scheme, such as is used to provide for proper handling of evidence in legal cases or to manifest waste handling procedures, to provide assurance that sample identification was correct. The sample tracking system employed sample custody forms, receipt forms, and storage forms. Every BB sample was assigned an identification number that was associated with the sample from the initial collection through the analysis and data workup. A record that correlated ELI identification numbers with a description of the corresponding sample was maintained to assist in sample and data management. The lists were updated as the need for changes became evident, such as when samples were split or errors in a description for a given ELI number were brought to ELI's attention.

3.1.1.2 ELI verified that sample custody forms were prepared for all samples taken during the BB testing. However, the forms were not always promptly maintained by some laboratories. For example, during the concentrated early periods of testing, OGC and SNL dedicated most of their manpower assets to timely completion of the testing requirements and felt that a level of documentation required for legal purposes was excessive for documenting the handling of lab samples derived from these investigative studies. ELI agrees to revise documentation forms for future tests to make them more appropriate for specific sample-handling situations.

3.1.1.3 Compliance audits by ELI verified that AWL and BCD used in-house tracking forms, but the remaining laboratories used alternative schemes. At LBL, samples were logged in, given a test sequence number, and cross-checked both manually and by computer to ensure accuracy. At SNL, the sample number and the date were logged into a notebook. At OGC, numbers were assigned each cylinder and entered into a logbook prior to being sent to the BB test site; upon return receipt of cylinders at OGC for analysis, dates were logged and numbers were checked for accountability. OGC also cleaned their cylinders immediately after analysis as part of the analytical routine. These

alternative, internal sample tracking systems were considered to be effective and to eliminate the need for separate forms for internal laboratory use on future phases of the OB/OD program.

3.1.1.4 During the BB study, if a sample was split for analysis or archiving reasons, ELI copied the collection report so that a duplicate form could accompany the additional sample; however, this created a potential for confusion when tracking and when compiling results since one distinct portion of the same sample carried an original form and the other a copy of the same form. In future phases of the OB/OD study, a new ELI number (possibly by appending a letter) will be given to split or composite samples, with reference made to the original source sample ELI number.

3.1.1.5 SNL personnel did not use receipt, custody, and storage forms to track the TNT blocks utilized in the BB detonations because the facility employed an alternative internal tracking procedure, which ELI judged satisfactory. ELI received copies of the original shipping documents, and collection reports were used for the samples of the TNT blocks that were taken for elemental (C,H,N) or trace analysis.

3.1.1.6 Sample custody forms were completed on both the soil and resin samples submitted to EPA for spiking and for use as audit samples. Collection and shipping forms were not employed when handling these sets of samples.

3.1.1.7 Although the recommended forms were not employed in all cases, ELI judged that this did not permanently affect proper data identification or quality.

3.1.2 Laboratory Logbooks

3.1.2.1 ELI compliance audits established that all laboratories employed some form of logbook as part of their internal sample-tracking procedures. Most labs (AWL, BCD, LBL, and SSL) used ELI numbers when logging in samples. A few assigned a unique internal lab identification number to each sample received: OGC used the numbers they had assigned to outgoing tanks, and SNL used a standard institution-wide sample numbering scheme.

3.1.2.1.1 Alpine West Laboratories. AWL maintained a project-specific sample logbook for recording sample receipt. The SFC-MS operator used a project-specific personal logbook to record extraction information, all samples injected, and details of instrument performance. Because the SFC-MS was not used for any other project, no additional instrument logbook was maintained, but all calibration printouts for the SFC-MS were put in a dedicated loose-leaf notebook. The temperature of the AWL freezer was reportedly checked monthly, but there was no documentation.

3.1.2.1.2 Battelle-Columbus Division. Each sample receipt was recorded on a separate line in a logbook, using the ELI number and sample description. The book number, line number, and ELI number became the in-house sample identification number. A form was used at the freezer to record, according to identification number, in/out times and sample volume taken. Up-to-date temperature-monitoring sheets were located at the freezer. Each worker maintained a project-specific personal logbook to enter data concerning the sample taken, the time, the volume injected into the GC-MS, etc. A logbook was used at the GC-MS for recording samples analyzed, analyst name, and instrument-related details.

3.1.2.1.3 Lawrence-Berkeley Laboratory. LBL employed a large logbook to record sample identification, instrument performance, QC results, and location of corresponding experimental data. Removable hard disks were used to store all experimental data, including sample numbers and calibration information. The logbook was written in pencil to allow neat corrections, but data integrity was not threatened because all data was stored electronically.

3.1.2.1.4 Oregon Graduate Center. OGC used personal logbooks to record tank identification and pertinent analytical information. Calibration information was stored with the corresponding analytical data. Accounting was not needed for long-term sample storage, because tanks were cleaned immediately after analysis.

3.1.2.1.5 Sandia National Laboratories. SNL used personal logbooks to record sample numbers, timing information, etc. A project-specific logbook was maintained for each individual instrument for recording information such as zero and span checks, calibration, and maintenance. Data from the individual notebooks was also recorded electronically, and real-time instruments directly produced electronically-recorded signals.

3.1.2.1.6 Sunset Laboratory. The principal investigator is the only worker at SSL. He used a personal logbook to record all information concerning analysis runs. In addition, the instrument calibration and analysis printout was stored electronically.

3.1.3 Written Procedures.

3.1.3.1 ELI performed compliance audits to verify that the workers in the BB experiments followed detailed written procedures. The investigative nature of the BB phase of the OB/OD project and the scheduling made it impractical for the various operators to develop, in advance, formal standing operating procedures (SOPs). As is common in such situations, letters of instruction (LOIs) were employed instead because approved changes in procedure needed to be allowed during the investigation's methodology-development periods. However, members of the TSC had to approve the proposed changes in key procedures before they could be implemented. The principal investigators documented or confirmed all procedures described in the LOIs to ensure that the LOIs adequately described the methodologies being used. That these procedures were developed and applied or supervised by truly nationally and internationally recognized experts was considered an important factor in ensuring achievement of high quality. Each LOI to be used during BB sample or data analysis was also reviewed for technical adequacy by the chairman of the TSC or by selected members. After suggested revisions were considered and incorporated, a copy was filed with ELI, which functioned as a clearinghouse, archiving agency, and auditor. The LOIs developed for the BB trials are included as Appendix B to this volume.

3.1.3.2 ELI compliance audits checked internal agency adherence to the versions of the LOIs that were current at the time of the site visit. Observations made during the visits relevant to LOIs are summarized below.

3.1.3.2.1 Alpine West Laboratory. At the time of the site visit (25 Jan 1989) AWL used acetonitrile to extract the filter samples, in accordance with the LOI. On 4 Feb 1989, the LOI was revised and methylene chloride was used to extract BB samples from the 31 January through 16 February 1989 trials. At the time of the visit, AWL workers were extracting all the samples (from preliminary BB trials) and sending one-half the extract to BCD for analysis. For the main series of BB trials, the samples analyzed at BCD were all extracted at BCD.

3.1.3.2.2 Battelle-Columbus Division. As specified in the LOI, a calibration standard was analyzed daily at the time of the site visit (28 December 1988). For the 21 January through 16 February series of trials, they used a moving average response factor based on the analysis of standards that bracketed, in time, a particular group of samples. All NCI-SIM results were based on the new procedure, included in Appendix B.

3.1.3.2.3 Lawrence-Berkeley Laboratory. LBL did not produce a LOI specifically for the BB work, but they provided a generic writeup of their EPA-approved procedure. The analyses were done according to a well-practiced routine, and no deviations from the written procedures were noted -- except that at the time of the visit (31 Jan 1989) data were being recorded on a 20 mByte Bernoulli disk instead of a 5 mByte disk.

3.1.3.2.4 Oregon Graduate Center. OGC had not provided a LOI by the time of the site visit (24 Jan 1989); thus, the audit focused on compliance with good laboratory practices (GLP). The procedures employed by the laboratory personnel appeared to be meticulously done.

3.1.3.2.5 Sandia National Laboratories. At the beginning of the BB trials, SNL provided a LOI that dealt with the real-time instruments. At the day of ELI's site visit (17 December 1987), these instruments were not in use. However, EPA's performance and systems audit on 6 to 8 February 1989 included real-time instruments. The analysis of HCN and NH₃ bubblers was observed by ELI, although LOIs had not been prepared because standard procedures (not provided to ELI) were followed. GLP appeared to have been applied.

3.1.3.2.6 Sunset Laboratory. The analyst routinely performs a very specialized, EPA-approved procedure he developed. The LOI accurately described his manipulations.

3.1.4 Data Management and Archiving

3.1.4.1 The raw data received for the project have been archived at Andrulis Research Corporation, 31 Potter Street, Salt Lake City, UT. These records will be stored for five years, at which time the Program Manager will decide their final disposition or storage. Final reports from

AWL, BCD, and SNL have been received. The work by LBL, OGC, and SSL has been included in the SNL report.

3.1.4.2 The QAPP called for all data to first come to ELI for QA review, reproduction, and archiving (originals), after which copies were to be disseminated for data reduction and analysis. As the project progressed, however, certain key data were forwarded (principally by SNL) directly to ANDRULIS Research Corporation, prior to being received by ELI, to expedite the data reduction process. The QAPP also stipulated that ELI check the data for quality and completeness before distribution. While ELI's staff did check the data for general adherence to proper record-keeping practices, technical examination of each datum item by ELI was not feasible, because of the quantity of data involved and since defects in individual datum often only become apparent as detailed calculations are performed on the whole data set. ANDRULIS Research Corporation's analysis staff therefore also functioned as technical data quality validators as data were reduced and analyzed. Andrulis identified discrepancies and brought them to the attention of ELI and the individual laboratories involved so that the issues could be resolved.

3.2 Sampling and Real Time Measurements

3.2.1 Sampling Accuracy

3.2.1.1 Samples containing the OB/OD detonation and combustion products must be carefully and properly handled if the sensitive analytical methods planned for use can be expected to yield high-quality component identification and quantification results. Since one purpose of the BB trials was to evaluate candidate sampling methods for potential use in future studies, checking and comparing sampling systems and results occupied a large proportion of the BB field and data analysis efforts.

3.2.1.2 The BB is an air-supported, rubber-coated fabric hemisphere with a radius of 7.6 m. A 5.5-m long x 2.1-m high x 2.5-m wide plywood airlock provides access to the building. Air pressure supplied by a blower supports the building. A number of sampling instruments normally installed on the SNL atmospheric research aircraft (which were proposed for use in later, outdoor large-scale OB/OD tests) were positioned both in the airlock and inside the chamber so that data derived from direct chamber air and indirect (tube- or probe-sampled) chamber air could be compared. The

comparison allows determining if inaccuracies were introduced by the 5-m long, 8-cm diameter aluminum tube that serves as the sampling probe for particulate and gas samples collected during flight.

3.2.1.3 Table 3.1 lists samplers and real-time instruments used in the BB airlock. Air from the aircraft sampling probe (which extended from the airlock into the BB) was routed through a pneumatically driven 10-cm diameter gate valve into a 1.5-m³ carbon-impregnated polyethylene (Velostat™) sampling bag. The bag, constructed of electrically-conductive plastic material in order to minimize wall loss of charged particles, filled with air from the chamber interior in approximately 40 s. Stainless steel sampling lines connected to the aircraft probe led to filters, vapor collection systems, and real-time gas monitors also located in the airlock. Particulates and semivolatile organic compounds were collected on two sampling trains (semi-VOST), consisting of a modified commercially-available sampling unit containing a prefired quartz filter followed by two cartridges containing XAD-2™ resin. (Preliminary trials evaluated Porapak™ resin, but this sorbant was rejected as the resin of choice for use on data-collecting trials because the flattened resin particles are subject to airflow-restricting packing.) The first XAD-2™ cartridge contained 65 g of resin, which was backed up by a cartridge containing 20 g of resin to recover any component that might pass through the first cartridge during the aspiration. Other filters connected to the bag outlet manifold included a Teflon™ filter, used for gravimetric analysis (for particulates) and XRF measurements (for elements), and a Nuclepore polycarbonate filter used for scanning electron microscopy (to examine particle morphology). Five carbon-vane pumps that supplied a total airflow of approximately 200 liters per minute provided air movement from the bag through the filter. Mass flow meters enabled calculating total airflows through the samplers. In-line Teflon™ filters were used with real-time instruments to prevent contamination of the instrument optics by particulates. Air input to these instruments was selected by a manual valve from either the main sampling probe or from the sampling bag. A differential mobility particle sizer to measure particle size distributions in the 0.01 to 0.5 μm particle diameter range. On selected test days, a continuous flame ionization detector and a photoionization detector provided an approximate measure of volatile hydrocarbon concentrations in near real-time. To determine the magnitude of sorption on the inside surface of

Table 3.1. Samplers and Real-Time Continuous Monitors Located in the SNL BangBox Airlock.

Number ^a	Instrument	Purpose	Model
1	CO ₂ gas analyzer	Real-time concentration	TECO 41 H
2	CO gas analyzer	Real-time concentration	TECO 48
3	SO ₂ gas analyzer	Real-time concentration	TECO 43
4	O ₃	Real-time concentration	TECO 49
5	NO _x gas analyzer ^b	Real-time concentration	CSI 1600
6	THC analyzer (FID)	Real-time concentration	Century OVA-108
7	THC analyzer (PID)	Real-time concentration	HNU Model PI-101
8	Differential mobility particle spectrometer	Particle sizes 0.01 to 0.5 μ m	TSI DMPS
9	Aerodynamic particle spect.	Particle sizes 0.5 to 15 μ m	TSI APS
10	Teflon TM filter sampler	Metals analysis	GAST Model 30
11	Polycarbonate filter sampler	Particle morphology	GAST Model 30
12	6-L Stainless Steel evacuated canisters (VOC)	Chamber atmosphere	OGC custom design
13	0.85-L Stainless Steel evacuated canisters (SF ₆)	Chamber Volume	OGC custom design

^aItems numbered 1 through 11 are covered in SNL LOI titled "Gas and Aerosol Instrument Calibration and Sampling Procedures".

Items 12 through 13 are covered in OGC LOI titled "VOC Collection Analysis System".

^bAlso analyzes for NO and NO₂ (by difference).

the bag, or the extent of offgassing from the bag material, OGC personnel collected grab-samples of air in electropolished, passivated 6-L evacuated stainless steel cylinders directly from the sampling duct and indirectly from the 1.5 m³ sampling bag. OGC subsequently assayed the contents of these cylinders for H₂, CO, CO₂ and C₁ - C₁₀ volatile hydrocarbon concentrations by gas chromatography (GC) with thermal conductivity, flame ionization (for tracer SF₆ analysis) and electron capture detectors. Samples collected and analyzed from the bag, which was connected to the aircraft sampling probe, were called "indirect" samples.

3.2.1.4 Table 3.2 lists the instrument systems used within the BB to collect so-called "direct" samples. Two laser particle spectrometers, normally installed and flown on the SNL aircraft were used to make particulate measurements in real-time. One of these probes (FSSP) is designed to incorporate true in-situ measurement principles and therefore requires no correction for particle transmission or sampling losses. Both the FSSP and ASASP probes provided records of total particle counts in 1-min intervals. A flash-lamp integrating nephelometer and a portable forward light-scattering particulate detector provided continuous measurement of particulate concentration inside the chamber during each test. Video cameras, recordings, and (on occasion) a high-speed (5,000 frames/s) camera provided photographic coverage of detonation and burn trials. Two 1-m diameter fans with approximate airflow rates of 250 m³/min rapidly mixed the contents of the building prior to collecting those samples intended to be representative of well-mixed (homogeneous) chamber air. For purposes of data reduction, samples collected from the chamber interior immediately after detonation or burn and prior to turning on the mixing fans are considered to have come from "nonhomogeneous" air, and are so identified in all data. The fan blade surfaces were also sampled to determine how much of a given analyte might have adhered to the blade.

3.2.1.5 Semivolatile and organic particulate species were collected directly from the chamber interior by two modified semi-VOST samplers operating at flow rates of approximately 100 L/min. (Standard hi-volume sampler blowers with no flow control were used as the air movers for these direct semi-VOST sampling units, but calibrated mass flow meters were installed in the lines to ascertain the exact flow rates.) The filter and cartridge units used to collect samples in these semi-VOST samplers were identical to those used in the airlock. The direct semi-VOST were checked independently using evacuated, passivated, electropolished 32-L stainless steel cylinders (during the trials of 31 January to 6 February 1989).

Table 3.2. Samplers and Real-Time Continuous Monitors Located Inside the BangBox Chamber.

Number ^a	Instrument	Purpose	Model
1	Aerosol spectrometer	Particle size distribution (0.1-3 μm)	PMS ASAP-100-x
2	Aerosol spectrometer	Particle size distribution (1-47 μm)	PMS FSSP-100-x
3	Integrating nephelometer	Particulate concentration	MRI 1550
4	Nephelometer	Particulate concentration	MIE RAM-1
5	Video camera	Photometric record	Sony
6	Fast frame camera (5000 frame/s)	Photometric record	Unknown
7	semi-VOST sampler ^b	Filter and resin samples of semivolatile-organics	GMW PS-1
8	Gas impinger (bubbler) ^c	HCN, NH ₃ , and HCl concentration	Gillian 113FS
9	Evacuated stainless steel tanks ^d	Volatile and semivolatile organics	OGC custom design

^aNumbers 1 through 4 are covered in SNL LOI titled "Gas and Aerosol Instrument Calibration and Sampling Procedures". Items 5 and 6 furnished through SNL Photo Documentation Division and were not covered under submitted LOI other than operational information. LOI for items 7 and 8 are not covered in LOI except for operational information (SNL LOI "Gas and Aerosol Instrument Calibration and Sampling Procedures"). Item 9 is covered in OGC LOI titled "VOC Collection Analysis System".

^bTwo samplers were utilized routinely. For composite propellant burn an additional two samplers were utilized.

^cTwo bubblers were used except for composite propellant burn when two additional bubblers (in series) were used to measure for HCl.

^dNine tanks were used except for test of 15 Feb 89 in which 27 tanks were used including three cryogenic tanks (immersed in liquid N₂).

3.2.1.6 Glass impinger bubblers filled with appropriate absorbing solutions were aspirated within the BB to collect ammonia, hydrogen cyanide, and (for the composite burn) hydrogen chloride that might be produced. Two bubblers in series were used to collect the HCl sample.

3.2.1.7 During the composite propellant burn trial, medium-volume air samplers (283 L/m) took duplicate samples on quartz fiber filters backed up by pre-cleaned polyurethane foam-filled cartridges for analysis for PCDDs and PCDFs by BCD. The analyses included determination of total hepta-, hexa-, penta-, and tetra-CDD and CDF congeners, as well as octa- and 2,3,7,8-tetra-CDD and -CDF concentrations.

3.2.1.8 The sample volumes required for the analytical methods to detect parts per billion (ppb) levels of combustion products had been estimated by the TSC during the planning sessions. Mass flow meters calibrated before the BB trials and after completion of the series were used to determine the volumes of BB air actually drawn through each sampler. On-site measurements of temperature and atmospheric pressure were employed to correct the observed sample volumes to the volumes at standard conditions.

3.2.2 Quality Control

3.2.2.1 Because of the breadth of the BB investigation, a single individual was not appointed as sampling coordinator. Members of the TSC made the initial decisions about methods and equipment, and maintained oversight of the sampling operations at the BB. Primary on-site responsibility for sample collection details was borne by each of the appropriate principal investigators (from SNL, OGC, and BCD), with assistance and guidance by members of the TSC (from ARC, BCD, BYU, and DPG).

3.2.2.2 Control of sampling quality during the BB trials (to minimize systematic and random errors) was maintained primarily by using calibrated flow devices and calibrated real-time continuous samplers, by using a redundancy of samplers, and by including the most accurate sampling systems as a basis for comparison.

3.2.2.3 EPA-QAD audited the performance of the real-time instruments used during the BB trials of 6 to 8 February 1989. The results of the audit, discussed in Sections 3.3.6 and 3.3.7, show that the real-time sampling-and-analysis instruments performed well.

3.2.3 Completeness of Sampling Effort

3.2.3.1 Because this was an investigative effort, the number and kind of samples taken and analyzed were expected to change from those originally specified in the test design plan (TDP). Increases in the number of samples were often approved by the TSC, so that the option of further investigation through additional sample analyses would be possible. Similarly, the taking and storage of a sample was generally relatively inexpensive, but some analyses were very costly; thus not all available samples were analyzed, especially if it could be determined, by review of preliminary data, that the probability of useful information was low. When decisions were made on-site to take additional samples, sample custody forms were initiated, but ELI made extra checks of logbooks, etc., to ensure that all such samples were appropriately accounted for.

3.2.3.2 A completeness-of-sampling value calculated for a given type of sampler exceeded 100 percent when additional samples of the type specified in the TDP were taken. Table 3.3 presents the numbers of each type of sample as specified in the TDP and the numbers of samples collected.

3.3 Chemical Analyses

3.3.1 Analytical Instruments

Table 3.4 lists the analytical instruments or techniques used by the individual laboratories to analyze BB test samples. (The real-time continuous monitors are covered in Table 3.1 and 3.2 page 3-8 and 3-10 respectively.) The respective LOIs are attached to this report as Appendix B.

Table 3.3. Completeness of Sampling Effort During "BangBox" Tests.

Sample Type	Numbers of Samples Specified in Test Plan	Numbers of Samples Taken of Those Specified in Test Plan	% of Samples Taken of Those Specified in Test Plan	Numbers of Additional Samples from Onsite Decisions	Numbers of Additional Samples Taken of Those from Onsite Decisions	% of Samples Taken of Those Specified by Onsite Decisions
Resin & Filter for SVOC* by SFC-MS	145	145	100	37	37	100
Resin & Filter for SVOC by GC-MS	53	53	100	92	92	100
32-L Tank for SVOC	9	9	100	8	8	100
Canister for VOC* by GD-FID	30	39	100	80	80	100
Canister for SF ₆	0	0	100	289	289	100
Filter for Elements by XRF	15	15	100	32	32	100
Filter for Carbon Species	24	24	100	20	20	100
Bubblers & Filters for Gases & Particulate	21	21	100	49	49	100

*Semivolatile organic compounds.

**Volatile organic compounds.

Table 3.4. Instruments and Methods Used to Analyze BB Trial Samples.

Lab	Instrument or Method	Type of Analyte
AWL	Lee Scientific Model 602 SFC Finnigan MAT INCOS 50 MS Varian Model 3400 GC Finnigan MAT 8430 MS	Semivolatile organic compounds
BCD	Finnigan TSQ-45 GC-MS Finnigan 4500 MS	Semivolatile organic compounds
LBL	X-Ray spectrometer for LBL design	Metals and nonmetals
OGC	Perkin Elmer 3920 GC Carle 211 M GC Shimadzu GC - Mini 2 Trace Reduction Gas Detector RGD2	Volatile organic compounds
SNL	(See Table 3.1 for real-time instruments)	Gases
SSL	Thermal-optical instrument of SSL design	Elemental and volatilizable carbon

3.3.2 Detection Limits

3.3.2.1 Although the analytical methods were chosen to satisfy each individual requirement of the BB project based on expert knowledge of instrument capability, the analyte diversity and the project's investigative nature dictated that the laboratories characterize the performance of their instruments and the adequacy of their procedures before commencing the analysis of BB samples. For example, it was necessary to determine the efficiency of extracting the analytes from the resin. (Results of extraction efficiency studies are discussed in Section 3.3.7.) To be able to calculate the lowest levels detectable in the BB trials, it was necessary to know the detection limits for the individual analytes for each analytical method. Tables 3.5a, 3.5b, and 3.5c present and define the detection limits for each of the analytical instruments or techniques.

3.3.2.1.1 Alpine West Laboratory. An instrument "limit of detection" was defined as the compound concentration that gave a signal/noise ratio of 3 when measuring the single compound in a solvent. These values ranged from 0.03 to 2.3 ng/mL, depending on the analyte. (This common index of the ability to resolve a signal from the background does not specifically address the ability to quantify at that level for real world samples containing other compounds causing interferences.)

3.3.2.1.2 Battelle-Columbus Division. A reported "limit of quantification" corresponds to the lowest concentration of standard used to prepare the calibration curve for an analyte. The values ranged from 4 to 60 ng/mL, depending on the analyte. The detection limits for the two chromatography-MS laboratories (AWL and BCD) were determined using different approaches even though both were analyzing semivolatile organics. In this case, where there was interest in comparing two analytical methods, a common inter-lab definition and measure of detection and quantification limits was desirable; however, none of the customary measures are free of arbitrary or erroneous assumptions. For example, EPA's Method Detection Limit presumes that the variance of the blank applies to the detection of low concentrations of analyte; the Haubaux-Vos method assumes that the response factor and variance are constant even in the region of the detection limit, the standard curve extrapolates to the origin, and the readings follow a Gaussian distribution even as zero quantity of analyte is approached. Furthermore, most methods for specifying a detection limit do not properly differentiate the ability to detect analyte and the ability to quantify analyte.

Table 3.5a Detection Limits for Chemical Analyses Used on BangBox Samples.

Analyte	Detection Limits (ng/mL)				
	AWL ^a SFC-MS ^c		AWL Lower Instrument Detection Limit	BCD ^b GC-MS ^d	BCD Lower Quantification Limit
	PI-EI-SIM ^e	CI-SIM ^f		CI-SIM	
2,4-Dinitrotoluene	17.6	0.07	S/N ^g = 3	4.0	Lowest standard used for calibration
2,6-Dinitrotoluene	19.3	0.05		4.0	
Dibenzofuran	21.2	0.31		60	
N-Nitrosodiphenylamine	196	0.12		60	
2-Aminonaphthalene	125				
2,4,6-Trinitrotoluene	20.4	0.06		4.0	
2-Nitronaphthalene	90.3	0.03		4.0	
4-Nitrophenol	93.2	0.30		33	
Benz[c]acridine	124			60	
Benz[a]anthracene	118	0.73		60	
1-Nitropyrene	91.8	1.30		4.0	
Benzo[a]pyrene	92.4	2.30		48	
Dibenz[a,h]anthracene	91.6			48	
1,6-Dinitropyrene	196			10	
Naphthalene		0.62			
1-Methylnaphthalene		0.62			
2-Methylnaphthalene		0.62			
1,3,5-Trinitrobenzene		0.12			
Biphenyl		1.15			
Phenanthrene		1.20			
Pyrene		0.83			
Phenol				30	
2-Naphthylamine				30	

^aAlpine West Laboratories.

^bBattelle-Columbus Division.

^cSupercritical fluid chromatography - mass spectrometry.

^dGas chromatography - mass spectrometry.

^ePositive ion, electron impact, selective-ion monitoring.

^fChemical ionization, selective-ion monitoring.

^gSignal-to-noise ratio.

Table 3.5b Detection Limits for Chemical Analyses Used on BangBox Samples (Cont'd).

Analyte	LBL ^a Detection Limits (ng/cm ²)	Instrument Method	Lower Detection Limit
Aluminum	150	X-ray microprobe; XRF ^b	3 standard deviations of repeated reading of standards
Silicon	40		
Sulfur	15		
Chlorine	12		
Potassium	6		
Calcium	5		
Titanium	30		
Vanadium	20		
Chromium	15		
Manganese	12		
Iron	12		
Nickel	6		
Copper	6		
Zinc	6		
Gallium	4		
Germanium	3		
Arsenic	3		
Selenium	2		
Bromine	2		
Rubidium	2		
Strontium	3		
Lead	7		
Zirconium	8		
Molybdenum	6		
Silver	5		
Cadmium	6		
Tin	8		
Antimony	8		
Iodine	12		
Barium	35		

^aLawrence-Berkeley Laboratory.

^bX-ray fluorescence.

Table 3.5c Detection Limits for Chemical Analyses Used on BangBox Samples (Cont'd).

Analyte	SNL ^a Detection Limits	Units	Instrument Method	Lower Detection Limit
Carbon dioxide	1.2	ppmv	TECO Model 41H	S/N = 2
Carbon monoxide	0.1	ppmv	TECO Model 48	
Sulfur dioxide	2	ppbv	TECO Model 43	
Ozone	5	ppbv	TECO Model 49	
Oxides of nitrogen	6	ppbv	CSI Model 1600	
Total hydrocarbons	2	ppmv	Century PVA-108	
Total hydrocarbons	0.1	ppmv	HNU Model PI-101	
Hydrogen cyanide	0.5	μg	CN. elect.; NIOSH 116	Literature
Hydrogen chloride	5.0	μg	Cl ⁻ elect.; NIOSH 115	
Ammonia	0.3	μg	Colorim.; NIOSH 205	
Sunset Laboratory detection limits				
Organic/elemental carbon speciation	0.3	μg/cm ²	Thermal optical instrument	3 Standard deviations of repeated readings of blank

^aSandia National Laboratories.

3.3.2.1.3 Lawrence-Berkeley Laboratory. LBL based the detection limits on three times the standard deviation observed from analyzing standards. These limits ranged from 2 to 150 ng/cm², depending on the element.

3.3.2.1.4 Oregon Graduate Center. The threshold of the GC-system integrators was the limiting factor in determining the lower detectable limit. Approximately 0.2 µg/m³ of a volatile hydrocarbon could be detected in air samples collected in 6-L canisters.

3.3.2.1.5 Sandia National Laboratories. SNL based the detection limits for gases on a signal-to-noise ratio of 2. As seen in Table 3.5c, the limits ranged from 2 ppbv to 2 ppmv, depending upon the particular real-time instrument.

3.3.2.1.6 Sunset Laboratory. SSL determined detection limits for analyses of organic and elemental carbon by performing many instrument blank analyses (analyses performed on a filter punch aliquot that was known to contain no carbon). The standard deviation of the blank was 0.2 to 0.3 µg carbon/cm² of filter.

3.3.3 Calibration Procedures

3.3.3.1 Because instrument readings must be correlated with analyte concentration through a valid response function, all of the instruments were fully calibrated prior to use, and calibration checks were run at least daily; some procedures included additional calibration checks during a day's run. ELI compliance audits verified that the laboratories performed their calibrations according to the method and schedule specified in their respective LOIs.

3.3.3.1.1 Alpine West Laboratory. AWL ran a full calibration at the start of the project to verify linearity. A mass calibration was performed daily. A standard containing the analytes was run at the beginning of each day to establish the response factors. A two-component internal standard was introduced into each extracted sample. They ran blanks containing the internal standards every third day.

3.3.3.1.2 Battelle-Columbus Division. BCD ran a five-point calibration covering three orders of magnitude of concentration to establish linearity of response relative to the included internal

standard. Calibration for quantification was performed before and after each group of samples (i.e., several times a day) using a standard containing single concentrations of each analyte. (The analytes that were included depended on whether negative or positive ion mode was to be employed for the samples.) BCD used a moving average response factor based on two analyses of the calibration standard. The MS was mass calibrated daily. An instrument blank and/or method (extraction and concentration) blank containing only internal standard was analyzed at least once each day.

3.3.3.1.3 Lawrence-Berkeley Laboratories. LBL ran a standard filter containing single concentrations of S, Cu, and Ag daily. Absolute calibrations had been previously determined. The daily values were used to normalize sample results for any small day-to-day variation in output intensity of the x-ray tube.

3.3.3.1.4 Oregon Graduate Center. OGC ran a three-point calibration standard (plus blank) in duplicate and determined response factors. At the beginning of each day, three analyses were made of a single point neohexane working standard. If the results fell within ± 2 percent, they used the average response to calibrate the analyses. If the three initial values exceed ± 2 percent, a fourth and fifth analysis was performed. A single point calibration check standard was included after every fourth sample to check for calibration drift. They added an internal standard to every fourth sample.

3.3.3.1.5 Sandia National Laboratories. SNL calibrated real-time instruments before and after a test with NBS standard gases, and made daily checks of zero, flow, and span. The SO₂ span was verified to be linear during EPA-QAD's Performance Audit.

3.3.3.1.6 Sunset Laboratory. SSL performed a multi-point calibration approximately every 100 samples. Single-point calibration checks were done every 30 samples. An internal calibration standard was introduced into every sample. They ran instrument blanks every 30 samples.

3.3.4 Quality Control

3.3.4.1 A single OB/OD program coordinator was not appointed for monitoring QC for the chemical analysis procedures. The conduct of the laboratory phases of the BB study was the responsibility of the respective principal investigators.

3.3.4.2 A variety of mechanisms was used to maintain quality control over the chemical analysis processes and/or systems. In addition to frequent calibration checks, as already noted, travel blanks (consisting of unexposed filters and resin cartridges), were placed with a group of test site samples as they were shipped. Their analysis result values were used as sampling media background levels and to help interpret the results of analyzing actual trial samples. Each laboratory employed method blanks to help evaluate the background level of target analytes that might be introduced with reagents used, by handling within the laboratory, or by other mechanisms. The values for method blanks were properly subtracted before reporting an observed concentration or quantity value for each actual trial sample. Each analytical laboratory LOI includes points related to the laboratory's internal QC program, as outlined below. Table 3.6 summarizes major features of each laboratory's internal QC procedures. During the compliance audit visits, ELI verified that the laboratories did follow the QC procedures specified in their LOIs and that good laboratory practices were followed.

3.3.4.2.1 Alpine West Laboratory. Dr. Christine Rouse, Dr. Karin Markedis, and Dr. Milton Lee were responsible for SFC-MS QC measures. If response factors from the daily standards differed from historical values by more than 10 percent, the instruments were recalibrated. If an internal standard differed from the known value by more than 10 percent, the MS system automatically rejected the value. Spiked control standards consisting of 300 pg of 1-nitronaphthalene-d, and 240 pg of 9-phenylanthracene were analyzed at least every other day. All samples were analyzed in duplicate to determine the precision of the derived concentration.

3.3.4.2.2 Battelle-Columbus Division. The institutional QA unit at BCD acted in an advisory role, and Dr. Lawrence Slivon, Project Director, was in charge of QC for the analysis of BB samples. At the beginning of each analysis day, and at intervals throughout the day, BCD analyzed a standard that contained all the target compounds plus an internal standard, along with blanks that contained the internal standard.

Table 3.6 Summary of Laboratory Calibration and Quality Control Procedures.

Lab	Calibration Standards	Internal Standard	Control Spikes	Other
AWL ^a	Beginning of each day.	Introduced into each extracted sample.	Spiked samples run every two days.	Instrument blanks; extraction efficiency
BCD ^b	Beginning of each day.	Introduced into each extracted sample.	Every 2-6 samples; end of every day.	Instrument blanks beginning of each day, every 2-6 samples; extraction efficiency.
LBL ^c	Run daily; contains S, Cu, and Ag.	NN ^d	Run daily; contains 19 elements.	Filter blank run with every tray of samples.
OGC ^e	Every 4th sample	Every 4th sample.	Daily	
SNL ^f	Calibrated with a known concentration of O ₃ , NO _x , CO, CO ₂ , and SO ₂ before and after test.	NN	NN	Daily check of zero, span, and flow.
SSL ^g	Introduced with each sample.	Introduced with each sample.	Known amount of carbon from sucrose standard solution.	Single-point check every 30 samples; instrument blanks every 30 samples.

^aAlpine West Laboratories.

^bBattelle-Columbus Division.

^cLawrence-Berkeley Laboratories.

^dNone.

^eOregon Graduate Center.

^fSandia National Laboratories.

^gSunset Laboratories.

3.3.4.2.3 Lawrence-Berkeley Laboratory. Dr. Robert Giaque was responsible for the QC program. Besides the daily calibration check, they ran a control filter and a blank filter each day. The control filter contained known quantities of 19 elements. The results from the control filter were used to verify the stability of the x-ray spectrometer system and the consistency of the overall analysis. The ratio of the observed values to the standard values for each of the 19 elements were plotted. The technical staff used three-sigma control limits as the criteria for rerunning the day's samples. In the previous two years of operation, deviations beyond three sigma limits only occurred following a major malfunction of the system.

3.3.4.2.4 Oregon Graduate Center. Dr. Reinhold Rasmussen managed the QC responsibilities. A calibration check standard was run after every fourth sample. An internal standard was added to every fourth sample. Humidified zero air certification served as the blank for the calibration/check process.

3.3.4.2.5 Sandia National Laboratories. The QC officer for the BB project was Mr. Wayne Einfeld. All instruments were calibrated, or zero and span checks were made prior to a trial. Flow checks were made immediately prior to and following each trial.

3.3.4.2.6 Sunset Laboratory. A single person (Robert Cary) performed all aspects of these analyses; there was no separate QC supervisor. He observed the automatic carbon spikes at the completion of each sample run. If the value did not fall within a certain specified range, it indicated that the unit was malfunctioning. If the single-point calibration standards (inserted every 30 samples) were not within $\pm 5\%$ of the known value, sections of the analysis equipment were checked, adjusted, repaired, or replaced, as needed.

3.3.4.3 The project's investigative nature and the high cost of running samples by SFC-MS and GC-MS necessitated some deviations from implementation of a classical QA/QC program; i.e., replicate sets of QC standards were not analyzed by most laboratories and QC charts were not maintained. (The amount of data required to set acceptability limits for QC charts is frequently not obtainable during short research runs; however, LBL and SSL did analyze their actual BB trial samples as a minor part of their normal workloads, and other labs could have based control limits on past performance with similar samples.) Nevertheless, even without classical, production-line QC, it is possible to extract performance data from readings for blanks, calibration standards, and

calibration checks, as well as from audit samples submitted by an outside agency. The results of the quality checks are discussed under Precision, Section 3.3.5, and Accuracy, Section 3.3.6, below.

3.3.5 Preventive and Remedial Maintenance

Checks of the logbooks verified that all laboratories performed preventive maintenance according to the equipment manufacturers' schedules. (E.g., every three months the manufacturer's service person performed a maintenance check on AWL's MS.) Any other required corrective maintenance was to be performed when the QC checks indicated a need or the instruments malfunctioned. Because of the importance of this project, BCD performed special cleanup and maintenance before commencing with the BB trial samples. AWL started with a newly-purchased SFC-MS.

3.3.6 Precision

The precision values obtained for each chemical measurement and the basis for the determinations are summarized in Tables 3.7a, 3.7b, 3.7c, and 3.7d.

3.3.6.1 Alpine West Laboratory. AWL obtained an analyte-dependent precision estimate ranging from 0.57 to 8.5 percent relative standard deviation (RSD) from two consecutive 1- μ L injections of duplicate portions of the same 1-mL sample concentrate.

3.3.6.2 Battelle-Columbus Division. Precision estimates, derived from duplicate injections of aliquots from 2-mL samples reinjected on different days, ranged from 7 to 14 percent RSD.

3.3.6.3 Lawrence-Berkeley Laboratory. LBL acquired three x-ray spectra, one for each excitation condition, for every sample. The precision varied from 0 to 12.8 percent RSD, depending on the element, except for Al and Br, which showed RSD's of 141% and 62.5%, respectively.

3.3.6.4 Sandia National Laboratories. Because SNL employed continuous-trace instruments to monitor real-time airborne concentrations, standard deviations were not determined. Most of the real-time instrument models have been declared as EPA "equivalent" instruments having met certain EPA requirements for precision capability. These values are listed in Table 3.7d.

Table 3.7a Precision for Analytical Procedures Used on the BangBox Samples.

Lab	Instrument Method	Analyte	Precision (% RSD ^a)	Basis of Determination
AWL	SFC/MS; CI-SIM ^b	2-Nitronaphthalene	6.8	Duplicate injections of TNT test samples (resins)
		2,4-Dinitrotoluene	1.5	
		2,6-Dinitrotoluene	11.7	
		4-Nitrophenol	NF ^c	
		Dibenzofuran	2.4	
		N-Nitrosodiphenylamine	2.3	
		Benz[a]anthracene	NF	
		Benzo[a]pyrene	NF	
		1-Nitropyrene	1.1	
		2,4,6-Trinitrotoluene	7.7	
		Naphthalene	5.2	
		1-Methylnaphthalene	7.5	
		2-Methylnaphthalene	7.5	
		1,3,5-Trinitrobenzene	3.9	
		Biphenyl	0.7	
		Phenanthrene	1.2	
		Pyrene	2.2	
		2,5-Diphenyloxazole	5.8	
		1,1,3-Trimethyl-3-phenylindane	9.5	
		2-Nitronaphthalene	1.5	Duplicate injections of TNT test samples (filter)
		2,4-Dinitrotoluene	2.0	
		2,6-Dinitrotoluene	5.3	
		4-Nitrophenol	NF	
		Dibenzofuran	8.5	
		N-Nitrosodiphenylamine	10.6	
		Benz[a]anthracene	NF	
		Benzo[a]pyrene	NF	
		1-Nitropyrene	NF	
		2,4,6-Trinitrotoluene	5.0	
		Naphthalene	1.0	
		1-Methylnaphthalene	NF	
		2-Methylnaphthalene	NF	
		1,3,5-Trinitrobenzene	0.9	
		Biphenyl	5.0	
		Phenanthrene	NF	
		Pyrene	NF	
		2,5-Diphenyloxazole	5.3	
		1,1,3-Trimethyl-3-phenylindane	11.7	

Table 3.7b Precision for Analytical Procedures Used on the BangBox Samples (Cont'd).

Lab	Instrument Method	Analyte	Precision (% RSD*)	Basis of Determination
AWL	SFC/MS; CI-SIM (cont'd)	2-Nitronaphthalene	7.6	Duplicate injections of TNT test samples (32-L tank)
		2,4-Dinitrotoluene	1.2	
		2,6-Dinitrotoluene	1.0	
		4-Nitrophenol	5.4	
		Dibenzofuran	4.4	
		N-Nitrosodiphenylamine	NF	
		Benz[a]anthracene	NF	
		Benzo[a]pyrene	NF	
		1-Nitropyrene	NF	
		2,4,6-Trinitrotoluene	2.9	
		Naphthalene	2.7	
		1-Methylnaphthalene	NF	
		2-Methylnaphthalene	NF	
		1,3,5-Trinitrobenzene	5.6	
		Biphenyl	NF	
		Phenanthrene	5.9	
		Pyrene	3.1	
		2,5-Diphenyloxazole	8.2	
		1,1,3-Trimethyl-3-phenylindane	12.9	
AWL	SFC/MS; CI-SIM	2-Nitronaphthalene	2.0	Duplicate injections of propellant test samples
		2,4-Dinitrotoluene	NF	
		2,6-Dinitrotoluene	NF	
		4-Nitrophenol	15.8	
		Dibenzofuran	0.9	
		N-Nitrosodiphenylamine	1.5	
		Naphthalene	2.7	
		4-Nitrosodiphenylamine	0.9	
		2-Nitrosodiphenylamine	2.7	
		4-Nitrodiphenylamine	5.1	
		Nitroglycerine	4.7	
		Resorcinol	4.1	
		Triacetin	4.2	
		Diphenylamine	2.0	
BCD	GC/MS; CI-SIM	4-Nitrophenol	14.0	Test samples run on two different days
		2,4,6-Trinitrotoluene	7.0	

Table 3.7c Precision for Analytical Procedures Used on the BangBox Samples (Cont'd).

Lab	Instrument Method	Analyte	Precision (% RSD*)	Basis of Determination
LBL	X-ray microprobe; XRF ^d	Aluminum	141.0	Control standard run on two different days.
		Silicon	2.11	
		Sulfur	0.46	
		Chlorine	0.00	
		Potassium	0.64	
		Calcium	0.52	
		Titanium	1.36	
		Vanadium	12.8	
		Chromium	6.67	
		Manganese	3.09	
		Iron	0.49	
		Nickel	0.39	
		Copper	12.50	
		Zinc	0.51	
		Gallium	7.07	
		Germanium	0.00	
		Arsenic	8.89	
		Selenium	0.00	
		Bromine	62.5	
		Rubidium	0.00	
		Strontium	1.34	
		Lead	0.32	
		Zirconium	4.82	
		Molybdenum	0.88	
		Silver	6.67	
		Cadmium	0.00	
		Tin	0.22	
		Antimony	2.12	
		Barium	0.96	
OGC	GC/FID*	Vinyl chloride	2.0	Replicate EPA audit samples; 3.2 to 4.2 ppb
		Bromomethane	4.0	
		Trichlorofluoromethane	2.0	
		Carbon tetrachloride	5.0	
		Methylene chloride	5.0	
		Chloroform	3.0	
		1,1,1-Trichloroethane	4.0	
		1,2-Dichloroethane	3.7	
		Benzene	9.0	
		Toluene	15.0	
		1,2-Dibromoethane	16.0	

Table 3.7d Precision for Analytical Procedures Used on BB Samples (Cont'd).

Lab	Instrument Method	Analyte	Precision (% RSD ^a)	Basis of Determination
OGC	GC/FID (cont'd)	Tetrachloroethylene	7.0	Certification of 0.226 ppmv standard.
		Chlorobenzene	18.0	
		o-Xylene	40.0	
		Trichloroethylene	4.0	
		1,2-dichloropropane	14.0	
		Ethylbenzene	32.0	
		Neohexane	2.4	
SNL	TECO model 41H model 4q1H	CO ₂	ND ^f	ND
	TECO Model 48	CO	5	EPA-Equiv ^g
	TECO Model 43	SO ₂	10	
	TECO Model 49	O ₃	10	
	CSI Model 160	NO _x	20	
	Century OVA-108	THC ^h	5	NN ⁱ
	HNU Model PI-101	THC	5	
SSL	Thermal-optical instrument	Organic carbon	5	Duplicate test samples
		Elemental carbon	5	

^aRelative standard deviation.

^bChemical ionization, selective-ion monitoring.

^cNot found in this particular sample matrix.

^dX-ray fluorescence spectrometer.

^eFlame ionization detector.

^fNot determined.

^gInstrument meets the EPA-equivalent method requirements in CFR Part 53.

^hTotal hydrocarbons.

ⁱNot known. (The THC instruments were used for qualitative indication only.)

3.3.6.5 Sunset Laboratory. SSL ran duplicate analyses on about ten percent of the samples. The RSD obtained from such duplicates has been approximately five percent.

3.3.7 Accuracy

3.3.7.1 The degree of accuracy of a chemical analysis measurement or value depends upon the effects of both random and systematic errors. The magnitude of the standard deviation (precision) has already been discussed. Frequently, specially designed, exhaustive studies are required to ascertain the magnitude of the systematic errors. If a standard is available whose true value is within satisfactory limits, a comparison of the value obtained from analysis of such a standard with the accepted value is normally taken as a measure of the accuracy of the procedure. (For example, the analysis of spiked blind samples prepared independently and from materials of a higher level of quality in the hierarchy of standards provides a determination of accuracy.) If a certified standard is not available, a common practice is to determine the percent recovery after adding a known quantity of the target or surrogate analyte (preferably from a source independent from the field sample). Percent recovery can be a less reliable determinant of accuracy, depending on the nature and effect of the sample matrix (whether the errors are additive or proportional), but it is widely used as an indication of the accuracy of a chemical analysis in the presence of potential interferences.

3.3.7.2 In assessing the percent recovery for the chemical analysis, the results reflect the steps involved in extraction, concentration, and analysis. Prior to the BB tests, when extraction procedures were being developed, EPA-QAD spiked the resins (provided by AWL), DPG soil samples, 6-L and 32-L canisters to prepare audit samples for use within AWL, BCD, and OGC. The resulting recoveries are in Appendix D, Enclosure 10.

3.3.7.2.1 Alpine West Laboratory. AWL determined the efficiency of extracting semivolatile organic analytes from XAD-2™ resins for both methylene chloride and acetonitrile. As detailed in Table 3.8a the values ranged from 62.1 to 103.4 percent, depending on analyte.

3.3.7.2.2 Battelle-Columbus Division. Methylene chloride extraction efficiencies ranged from 92 percent to 101 percent for removing semivolatile organic analytes from resin, as seen in Table 3.8b.

3.3.7.2.3 Lawrence-Berkeley Laboratory. LBL substantiated calibration procedures through the analysis of National Bureau of Standards (now NIST) Reference Standards Materials (SRMS), United States Geological Reference Materials, and Reference Materials from the National Institute of Environmental Studies of Japan. Estimates of the total calibration errors ranged from one to three percent for elements having atomic weights of 16 or greater, and ten and twenty percent, respectively, for the lighter elements Si and Al (Table 3.8b). LBL has participated in a number of round-robin studies and their results have always been among the best.

3.3.7.2.4 Oregon Graduate Center. OGC analyzed EPA-QAD audit samples of benzene in 6-L VOC canisters. The recovery ranged from 89 to 120 percent for individual results (Table 3.8b). The periodic certification of the neohexane daily working standard has yielded a maximum difference of -3.5 percent with a maximum RSD of 4 percent over the last five years.

3.3.7.2.5 Sandia National Laboratories. SNL obtained accuracy figures from the results of an EPA Performance Audit conducted during the BB study. These data indicated differences ranging from 7.0 to +12.7 percent of the known values of the gases audited (Table 3.8b and 3.8c). These values were within the target limits of ± 15 percent.

Table 3.8a Accuracy of Analytical Procedures Used on BangBox Samples.

Lab	Analytical Procedure	Analyte	Accuracy Determination	Known Concentration	Percent Different
AWL ^a	SFC/MS; CI-SIM ^b	2,6-Dinitrotoluene	Lab-spiked resin; extraction, evaporation, and analysis recovery	50 ng/ μ L	-1.3
		2,4-Dinitrotoluene			1.3
		2-Aminonaphthalene			-13.8
		2,4,6-Trinitrotoluene			-0.9
		2-Nitronaphthalene			3.4
		4-Nitrophenol			-9.9
		Benz[a]acridine			-6.4
		1-Nitropyrene			-7.9
		Benz[a]anthracene			-5.9
		Dibenz[a,h]anthracene			-11.7
		1,6-Dinitropyrene			-27.7
		2,6-Dinitrotoluene		0.5 ng/ μ L	-3.8
		2,4-Dinitrotoluene			-0.9
		2-Aminonaphthalene			-14.9
		2,4,6-Trinitrotoluene			1.4
		2-Nitronaphthalene			0.9
		4-Nitrophenol			-13.8
		Benz[a]acridine			-8.8
		1-Nitropyrene			-7.9
		Benz[a]anthracene			-8.4
		Dibenz[a,h]anthracene			-10.6
		1,6-Dinitropyrene			-37.9

Table 3.8b Accuracy of Analytical Procedures Used on BangBox Samples (Cont'd).

Lab	Analytical Procedure	Analyte	Accuracy Determination	Known Concentration	Percent Different (or actual)
BCD ^c	GC/MS; CI-SIM	2,6-Dinitrotoluene	Lab-spiked	20 ng/mL	-2.0
		4-Nitrophenol	resin; extraction, evaporation, and analysis recovery	50 ng/mL	-4.0
		2,4-Dinitrophenol		20 ng/mL	1.0
		2-Nitronaphthalene			0.0
		2,4,6-Trinitrotoluene			-3.0
		1-Nitropyrene		50 ng/mL	0.0
		1,6-dinitropyrene	-8.0		
LBL ^d	XRF ^e	Most Metals	NIST ^f standards; analysis recovery	50 to 150 $\mu\text{g}/\text{cm}^2$	± 3
OGC ^g	GC/FID ^h	Neohexane	NBS-SRM ⁱ benzene; analysis recovery	0.254	0.0
		Benzene	NBS-SRM propane; analysis recovery	2.87	-0.9
			EPA ^j audit; analysis recovery	0.9 ppbv	0.0
				0.9 ppbv	-11.0
				1.0 ppbv	10.0
				1.0 ppbv	20.0
				2.5 ppbv	16.0
				2.5 ppbv	4.0
				3.2 ppbv	13.0
		3.2 ppbv	6.7		
SNL ^k	Real Time	CO ₂	EPA audit; Analyzer analysis recovery	350 ppb	4.3
					3.8
					-1.4
					-0.6
		CO		36.5 ppm	4.1
				36.5 ppm	3.8
				6.2 ppm	0.0
				6.2 ppm	0.0
		SO ₂		422 ppb	0.0
				203 ppb	-0.25
				103 ppb	0.78
				61 ppb	1.5
				0 ppb	(-3.0)

Table 3.8c Accuracy of Analytical Procedures Used on BB Samples (Cont'd).

Lab	Analytical Procedure	Analyte	Accuracy Determination	Known Concentration	Percent Different (or actual)
SNL (cont'd)	Real time	O ₃	EPA Audit; Analyzers analysis recovery	199.6	-4.5
				99.9 ppb	5.4
				70.2 ppb	-6.5
				30.0 ppb	-6.6
				15.3 ppb	4.5
				0.0 ppb	(3.8 ppb)
		NO		1020 ppb	2.3
				855 ppb	4.3
				620 ppb	6.0
				430 ppb	10.0
				0.0 ppb	(76.2 ppb)
				NO ₂	420 ppb
		281 ppb			6.8
		0.0 ppb			(-2 ppb)
		NO _x			1020 ppb
				820 ppb	6.3
				620 ppb	10.0
				430 ppb	12.7
				0 ppb	(87 ppb)

Table 3.8d

Accuracy of Analytical Procedures Used on BangBox Samples (Cont'd)

Lab	Analytical Procedure	Analyte	Accuracy Determination	Known Concentration	Percent Different (or actual)
SNL (cont'd)	semi-VOST #1	Semivolatile Organic	EPA flow audit	98.8 L/min	8.3
	semi-VOST #2			103.3 L/min	7.3
	semi-VOST #3			67.2 L/min	-6.4
	semi-VOST #4			71.6 L/MIN	-9.2
	Bubbler #7	Toxic gas		1.99 L/min	-2.5
	Bubbler #9			1.85 L/min	-2.2
	Bag; Teflon XRF sampler	Metals and nonmetals		52.8 L/min	-1.7
	Bag; SEM sampler	Particulates		15.3 L/min	1.3
	BB; SEM sampler			2.87 L/min	0.4
SSL	Thermal optical instrument	Carbon	Sucrose lab standard analysis recovery	not given	±5

^aAlpine West Laboratory.

^bChemical ionization, selective-ion monitoring.

^cBatelle-Columbus Division.

^dLawrence-Berkeley Laboratories.

^eX-ray fluorescence spectrometer.

^fNational Institute of Science and Technology.

^gOregon Graduate Center.

^hGas chromatograph/flame ionization detector.

ⁱNational Bureau of Standards (now National Institute of Science and Technology) Standard Reference Material.

^jU.S. Environmental Protection Agency.

^kSandia National Laboratories.

^lSunset Laboratory

3.3.7.2.6 Sunset Laboratory. SSL estimated the accuracy for total carbon determination by analyzing the results of thermal treatment of sucrose standards. These means were within 5 percent of the known values (Table 3.8d). Speciation into organic carbon and elemental carbon was within 5 to 10 percent, depending on their relative amounts on the quartz-fiber filter.

3.3.7.3 Always of concern is whether or not analysis procedures are adequately free of interferences for the samples being analyzed. This was recognized by the TSC during the early planning meetings and was the principal reason for choosing the particular analytical methods employed for the assay of BB samples. The MS is one of the most sensitive detectors available. Combining MS with the separation capabilities of a GC or SFC chromatograph virtually eliminates the possibility that an interference will cause a false negative. Whenever solutions are analyzed by a highly sensitive analytical technique, the possibility of false positives from contamination can become extreme. An examination of the results of analysis of solutions of the semivolatile organic compounds indicates that false positives (or contamination) appear to be quite common for certain analytes -- probably a result of frequent occurrence of these chemicals in the modern environment. The low probability of false negatives and the high probability of false positives means that the concentrations and emission factors reported for most of the semivolatile organic compounds in OB/OD clouds are significantly safe-sided (i.e., Type II errors are minimized; the β risk is low).

3.3.8 Completeness of Chemical Analysis Effort

Because of the BB study's investigative nature, the number of samples to be collected and analyzed changed during the course of the work. Table 3.3 cites the number of samples of various types originally projected to be taken for analysis by the various laboratories and procedures. During the actual on-site conduct of testing, several management and technical decisions required collecting additional samples for analysis. For example, it was decided to collect and analyze a number of special samples to measure any PCDDs and PCDFs produced by the composite propellant burn. Similarly, two new trials were executed: (a) one to assess the ability of rigid aqueous foam to serve as a soil surrogate in chamber OB/OD trials, and (b) the other to provide an air sample with high potential to show measurable concentrations of OB/OD semivolatile combustion products by collecting emittants from eight successive shots of TNT in the same samplers. Once partial, preliminary assay results were available, management decisions eliminated the analysis of the

remainders of certain sets of samples due to the very low probability of finding specific analytes. Thus, these types of decisions led to laboratories being responsible for analyzing a different total number of samples than was originally outlined, so it was not meaningful to calculate a completeness for the number of samples analyzed. The target for completeness was 75 percent.

3.4 Data Reduction

3.4.1 Validity of Calculations.

The BB investigation produced a wide variety of measurement data that required a corresponding diversity of data reduction techniques. The use of computer spreadsheets, combined with manual spot-checking of individual entries and results to verify correctness of spreadsheet formulas and other manipulations, minimized calculational errors. During the course of data evaluation and analyses, the data analyst performed an informal data validation activity when observing the general pattern of data values and the presence of possible outliers.

3.4.2 Completeness of Data Reduction Effort

All of the experimental data (either raw or processed) has been received by ANDRULIS Research Corp for statistical analysis, for making derivative calculations, and for inclusion in the final report. Andrulis has not performed all possible statistical analyses, but has completed all analyses judged to be necessary to attain current data analysis goals.

SECTION 4. CONCLUSIONS

4.1 Objective

The objective of this part of the BB study was to identify and/or develop appropriate program specific QA/QC procedures.

4.1.1 Sample Tracking

The sample tracking procedures using sample custody forms, receipt forms, and storage forms resulted in identification procedures that provided chain-of-custody (identification) audit trail from sample preparation to the assay results. This tracking system will provide a credible method for field testing phase of the OB/OD study.

4.1.2 Audit Assurance System Audits

The systems audits based on the test design plan, LOI's, and good laboratory procedures produced recommendations to the individual participating organizations that resulted in revised LOI's that fully reflected procedures being used. The LOI's generated and revised during the BB test series will provide analytical procedures that can be used in the field testing phase of the OB/OD study.

4.1.3 Quality Assurance Performance Audits

The performance audits were based on EPA spiked samples and in-house controls using known levels of analytes.

4.1.3.1 Volatile Organics

All compounds in the audit mixture spiked in the 6-L canisters were correctly identified. There is a tendency of less volatile compounds to exhibit a pattern of decreased recovery. The problem of decreased recovery was resolved between OGC and EPA when moisturized air was used in the spiking procedure. The 6-L canister can be expected to give accurate and precise assessments when

spiking procedure. The 6-L canister can be expected to give accurate and precise assessments when used to samples volatile (C_1 to C_{10}) organic compounds.

4.1.3.2 Semivolatile Organics

4.1.3.2.1 The spiked resin analyses were declared invalid after interferences were noted by BCD in the analyses. Further investigation revealed that the resins were subjected to over heating during the cleaning phase and prior to spiking. The spiking was not repeated since the resin filled cartridge would not be used in the followon OB/OD field tests.

4.1.3.2.2 The 32-L tanks gave results that were predominately low in recovery, probably indicating incomplete extraction of compounds from the tank.

4.1.3.2.3 The spiking solutions were analyzed by both the GC/MS and the SFC/MS methods. The GC/MS gave satisfactory results except for the high value reported for 4-Nitrophenol. The SFC/MS was significantly lower for three of the four compounds reported.

4.1.3.2.4 A percent recovery factor will be developed for each semivolatile compound to correct for the extraction, concentration, and analysis loss of analyte.

4.1.3.3 Continuous Gas Monitors

The monitors were challenged with known concentrations of SO_2 , O_3 , NO, NO_2 , CO, and CO_2 . The performance audits results were excellent with results falling well within the target limits of ± 10 percent of the audit concentrations.

4.1.3.4 Other Performance Audits

All laboratories had their in-house performance audits which were deemed adequate by the QA/QC audit reviews.

SECTION 5 . RECOMMENDATIONS

5.1 The diversity of procedures employed and the extreme sensitivity required introduced special problems for QA/QC. To help overcome some deficiencies so as to improve the quality of subsequent OB/OD studies, the following comments are offered:

5.1.1 Because of the high probability of false positives occurring in trace chemical analyses when dilute air samples are extracted, concentrated, and analyzed by extremely sensitive analytical methods, at least two travel blanks of each appropriate kind should be included with the field samples sent to labs from each trial, in spite of the high per-sample cost of some analyses. These travel blanks should be processed in the same manner and simultaneously with the field samples.

5.1.2 Uniform methods of determining and reporting lower detection limits should be employed. Lower limits for quantitation should take into account the precision required for the data. (Precision often deteriorates with decreasing concentration, and the point at which precision becomes unsatisfactory depends on the data usage requirements).

5.1.3 A predetermined number of replicate assays should be run on different days, and these should include sample preparation steps. This will allow the derivation of a more valid estimate of experimental error for the overall chemical analysis process.

5.1.4 All QA and QC data from the BB phase of the study should be statistically analyzed to establish more realistic and achievable acceptability limits for subsequent phases.

5.1.5 Any changes in sampling, chemical analyses, or data reduction and statistical analysis procedures from that specified in the corresponding LOI should be documented in formal revision of the LOI which is then distributed to all concerned.

5.1.6 Any changes in sampling or analysis plans should be documented in formal addenda and/or revisions to the TDP; these revisions should be quickly approved and circulated to all personnel concerned.

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APPENDIX A. BANGBOX TEST SERIES - TEST DESIGN PLAN

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**HEADQUARTERS
U.S. ARMY ARMAMENT,
MUNITIONS AND CHEMICAL COMMAND**

**DEVELOPMENT OF
METHODOLOGY AND TECHNOLOGY
FOR
IDENTIFYING AND QUANTIFYING
EMISSION PRODUCTS
FROM
OPEN BURNING AND OPEN DETONATION
THERMAL TREATMENT METHODS.**

***BANGBOX TEST SERIES
TEST DESIGN PLAN***

SEPTEMBER 1988

Maintenance Management Division
Demilitarization and Technology Branch
Rock Island, Illinois 61299-6000
DSN: 793-3980/5534
Commercial: 309-782-3980/5534



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SECTION 1. INTRODUCTION AND SCOPE

1.1. Background

1.1.1. The manufacture and procurement of conventional ammunition and ordnance within the Department of Defense results in the generation of explosive residue, propellants, and munitions that must be demilitarized or destroyed as they become unsafe, are declared excess, are classified obsolete, or are determined to be uneconomical to maintain or repair. Demilitarization or destruction is a necessary and final step in the life cycle management of these items.

1.1.2. Assets requiring demilitarization are managed in the Special Defense Property Disposal Account (SDPDA), known within DoD as the "Demil Account." This account is an assets tracking system which ensures, among other factors, that appropriate final disposition considerations are made. Assignments of munitions and ordnance to the SDPDA or their classification as currently unserviceable, do not necessarily result in these items being demilitarized. They can be, and are, included in the foreign military sales program, the recycle program, and/or are reworked to meet original or alternative specifications. Therefore, an assignment of any ammunition or ordnance item to this account does not constitute a designation of such items as waste.

1.1.3. For those items within the Demil Account that are designated for immediate disposal, the treatment method commonly employed has been by open burning or open detonation (OB/OD).

1.1.4. The increasing requirements for data on explosive combustion products to support applications for State/Federal permits for continuation of OB/OD programs on many military installations present a critical need for a test program to collect these basic data.

1.1.5. Although there are limited data available from past studies on the generation of particulates and criteria pollutants from OB/OD operations, there are few data on the levels of emissions that result from incomplete combustion. These compounds will be difficult to collect and identify. Thus, prior to conduct of full-scale OB/OD field testing operations involving thousands of pounds of the explosive material in 3QFY89 at U.S. Army Dugway Proving Ground (DPG), Utah, a limited number of small-scale explosive detonation and propellant burning trials will be conducted within

a confined and controlled environment to characterize trace organic compound emissions, validate sampling and analysis procedures, and to establish quality assurance/quality control (QA/QC) procedures for instrument operation for the remainder (full scale field testing) of the OB/OD program. The test chamber to be used to provide this confined and controlled environment is operated by Sandia National Laboratories (SNL), Kirtland AFB, New Mexico, and is known as the "BangBox" (BB).

1.2. Objectives

The purpose of this test is to verify and validate OB/OD test technology including:

1.2.1. Instruments, sampling equipment, and procedures that will subsequently be used aboard a fixed-wing aircraft (FWAC) on full-scale field trials;

1.2.2. Procedures for transport and storage of sample specimens;

1.2.3. Sample analysis techniques;

1.2.4. QA/QC procedures; and

1.2.5. Cloud characterization (emission products).

1.3. Scope

1.3.1. This plan outlines concepts, equipment, and procedures to be applied in conduct of these preliminary tests, which will be conducted at SNL from 28 November 1988 through 17 February 1989.

1.3.2. The BB characterization trials have been structured to provide a fundamental understanding of what occurs within the BB when known materials (such as CO₂ and SF₆) are released, so that analyses of data from generation of explosive combustion products on subsequent trials within this facility will not be influenced by variables solely related to the properties of the BB. In addition, data from these trials will be used to evaluate and validate instruments and procedures that are candidates for use in full-scale OB/OD field testing.

1.3.3. A progressive series of subtests will determine:

1.3.3.1. The BB's leak rates with the blower on and the blower off;

1.3.3.2. The time to achieve gaseous homogeneity within the BB after release of a defined gaseous additive (SF₆); and,

1.3.3.3. Capabilities of OB/OD equipment and procedures to accurately capture and characterize representative samples of explosion emissions (particularly trace organic combustion products) following detonation of small explosive charges and propellant emissions following burning of small amounts of propellants.

1.4. Description of Material

1.4.1. Detonation of a 0.5-lb (227-g) block of trinitrotoluene (TNT) will generate a cloud of gaseous and aerosolized combustion products. Since TNT generates relatively large quantities of emissions and small amounts of TNT detonate less efficiently than larger amounts, a small-scale chamber test will provide the worst-case scenario (in terms of the generation of the potential pollutants and emissions of interest) expected during above-ground, large-scale OB/OD operations. This charge consists of pressed TNT grains ranging in size from -35 to +65 mesh. Initiation of the TNT block will be by one #6 blasting cap consisting of lead azide and hexamethylenetrinitramine (RDX) enclosed in an aluminum alloy casing equipped with two #16 insulated copper/tinned wires.

1.4.2. Burning of a 1.0-lb (454-g) sample of propellant will generate a cloud of gaseous and aerosolized combustion products. Two different propellant formulations have been selected for testing. The composition of these propellant samples are given in Tables 1.1 and 1.2. Generally, propellants contain more ingredients than explosives, making the prediction of combustion products somewhat more difficult. Careful selection of analytes, however, should permit accurate prediction of environmental impact. The traditional types of propellants contain the basic ingredients of nitrocellulose and nitroglycerin, and sometimes nitroguanidine. Composite propellants are composed of a rubber-based binder, such as polybutadiene, with an oxidizer, such as ammonium perchlorate, as basic ingredients. Additionally, many chemicals are used in small amounts for reasons such stability, burning rate modification, physical property enhancement, or as a processing aid. The propellant samples will be ignited with an electric match.

Table 1.1 Composition of NOSIH-AA-2 Propellant.

Ingredient	Weight (%)	Carbon Fraction (%)
Nitrocellulose	51.0	27.0
Nitroglycerin	38.6	15.9
Triacetin	2.7	49.5
Di-normal-propyl-adipate	1.6	62.6
2-Nitrodiphenylamine	2.0	67.3
Lead salicylate	1.5	34.9
Lead β resorcyate	0.5	32.8
Monobasic copper salicylate	2.0	49.8
Candelilla wax	0.1	85.2
Ethylcellulose (added)	5.9	58.5

Table 1.2 NOSIH-EC Propellant Composition.

Component	Weight Fraction %	Carbon Fraction %
Hydroxyl-terminated polybutadiene (R45M)	8.015	88.8
2,2-Methylene bis(4-methyl)-6-t Butyl phenol (AO 2246)	0.200	81.1
Diethyl sebacate (DOS)	4.500	73.2
Phenyl di-isodecyl phosphite	0.200	71.2
5-Ethyl-1,3-diglycidyl-5-methyl hydantoin diepoxide (XU-238)	0.300	56.7
Aluminum oxide	1.000	0
Carbon black	0.100	100.0
Ferric acetylacetonate (FeAA)	0.005	51.0
Ammonium perchlorate	85.000	0
Diethylene triamine (DETA)	0.080	46.6
Isophorone di-isocyanate	0.600	64.4

1.5. Test Facility

The BB is an air-supported hemisphere with an 7.6-m radius and a nominal volume of 927 m³. It is constructed of plastic-coated nylon fabric fastened to a concrete pad and inflated by a squirrel-cage low-pressure blower injecting air into the chamber (interior). The BB gradually deflates and loses its structural rigidity once the blower is shut off. Access to the BB chamber is through a 3- x 3- x 3-m airlock with 2- x 2-m plywood doors at each end. Interior wall areas adjacent to the door permit passage of electrical wires, flexible tubes, and small-diameter rigid pipes. The pressure differential between the BB chamber and the airlock precludes fresh air from entering the chamber, other than from the inflation blower.

1.6. Detection/Sampling Instrumentation and Collection Devices

1.6.1. Particulate Detectors and Samplers

1.6.1.1. TSI differential mobility particle sizer (DMPS) system: 0.01- to 0.5- μ m diameter.

1.6.1.2. TSI aerodynamic particle sizer (APS) system: 0.5- to 15- μ m diameter.

1.6.1.3. Teflon™ filter: 47-mm diameter, 2- μ m pore size; Membrana Inc.

1.6.1.4. Nuclepore™ filter: 0.2- μ m pore size, 47-mm diameter, Nuclepore™ #181106, to collect particulate samples for characterization by scanning electron microscopy (SEM).

1.6.1.5. Aerosol probe: 0.15- to 3.0- μ m diameter, PMS Active Scattering Aerosol Spectrometer Probe (ASASP)-100X.

1.6.1.6. Aerosol probe: 2- to 47- μ m diameter, PMS Forward Scattering Spectrometer Probe (FSSP)-100X.

1.6.1.7. Integrating nephelometer: MRI model 1550.

1.6.2. Total hydrocarbon (THC) detector.

Photoionization detectors (PID): ultraviolet (UV) lamp, 0.1 ppm detection limit, HNV instruments model PI101.

1.6.3. Gas Analyzers

1.6.3.1. Gas filter correlation CO₂ analyzer, TECO Model 41H.

1.6.3.2. Gas filter correlation CO analyzer, TECO Model 48.

1.6.3.3. Pulsed fluorescence SO₂ analyzer, TECO Model 43.

1.6.3.4. UV photometric O₃ analyzer, TECO Model 49.

1.6.3.5. Chemiluminescent nitrogen oxides (NO_x) analyzer, CSI Model 1600.

1.6.3.6. SF₆ analyzer, Shimadzu gas chromatograph (GC) with electron capture detector (ECD).

1.6.3.7. Bubblers for HCN and NH₃.

1.6.4. Trace organic material samplers

1.6.4.1. Resin cartridge train (Andersen PS-1), XAD-2™, 65-g cartridge followed by 20-g cartridge (Figure 1.1).

1.6.4.2. Quartz filters.

1.6.5. Samplers for metals: Teflon™ filters, 47-mm diameter, 2-μm pore size; Membrana, Inc.

1.6.6. Collectors - Velostat™ bag, 1 m³.

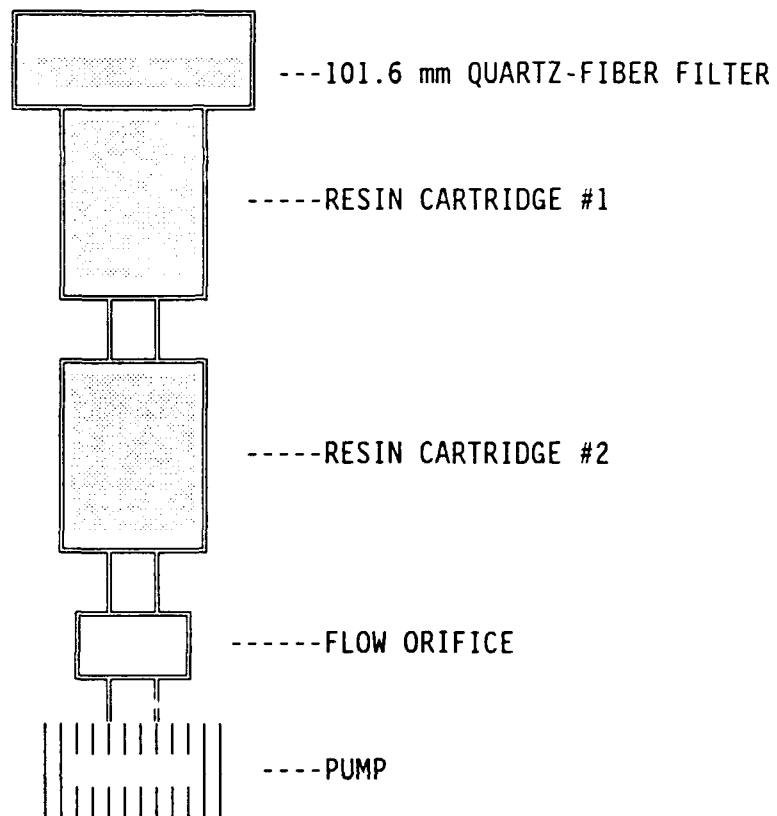


Figure 1.1 Andersen Model PS-1 Semivolatile Organic Sampling Train (VOST) Detail.

1.6.6.1. Stainless steel (SS), canisters 6 L.

1.6.6.2. SS tanks, 32 L.

1.6.7. Ancillary Equipment

1.6.7.1. Two mixing fans, 8,600 cfm, Granger Electric.

1.6.7.2. Color video camera.

1.6.7.3. Camera, high speed (10,000 frames/s).

1.6.7.4. Pumps (2), for semivolatile organic trains (VOST), Carbon Vane.

1.6.7.5. Flowmeters (6), for resin trains and non-organic sample filters, Matheson Model SEF-1454.

1.6.7.6. Dew point: EGG Model 880 chilled mirror hygrometer.

1.7. Detector, Sampler, Collector and Ancillary Equipment Siting

1.7.1. BB airlock: Instruments listed in paragraphs 1.6.1.1 thru 1.6.1.4, 1.6.2, 1.6.3.1 thru 1.6.3.6, 1.6.4, 1.6.5, and 1.6.6. Instruments and equipment will be arranged as in Figure 1.2.

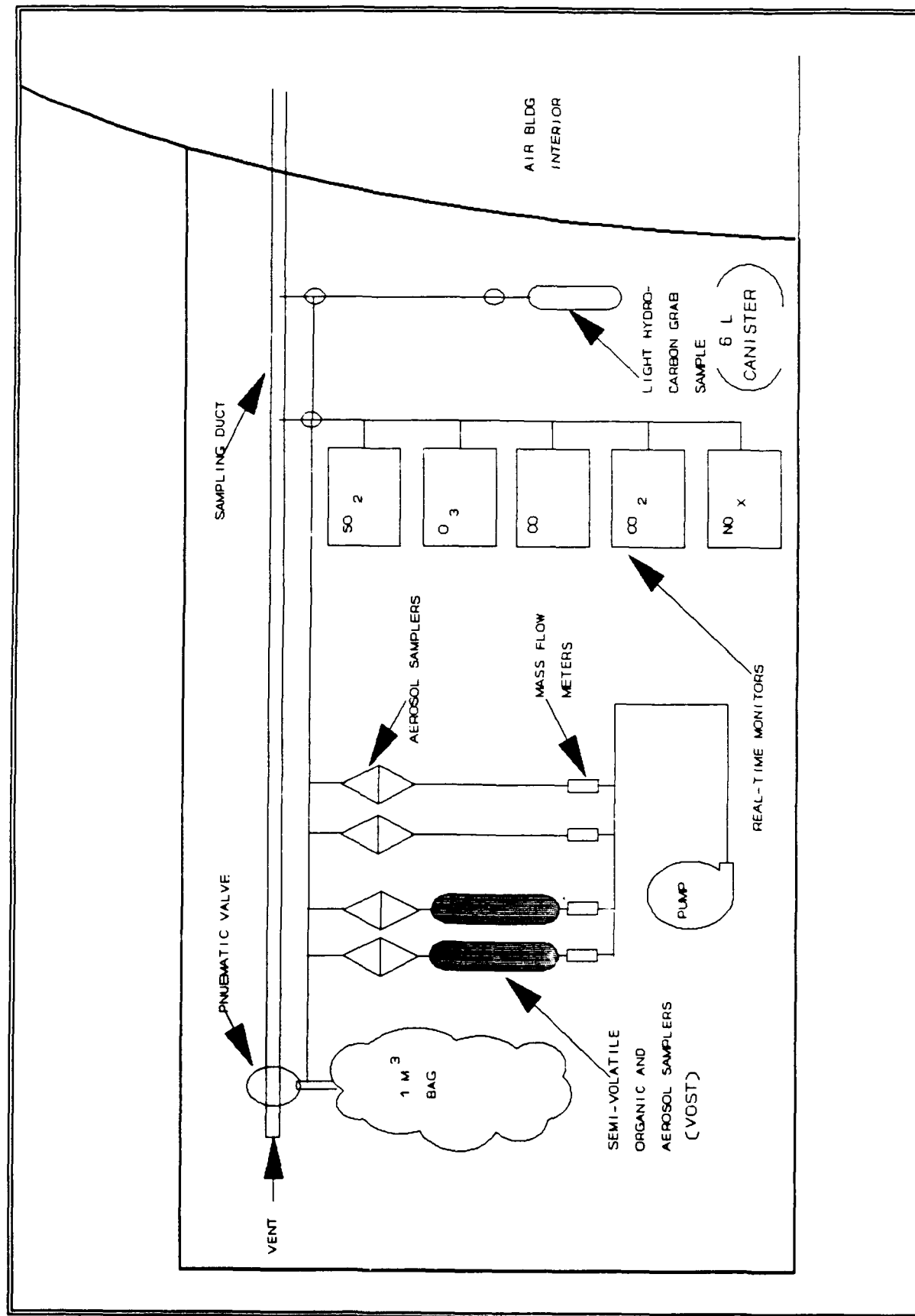


Figure 4.1 BangBox Airlock Instrumentation and Sampling Equipment.

1.7.2. BB chamber: Instruments listed in paragraphs 1.6.1.4 thru 1.6.1.7, 1.6.3.7, 1.6.4, and 1.6.5, Instruments and equipment will be arranged as in Figure 1.3.

1.8. Responsibilities.

1.8.1. DPG: Providing 15 soil samples from previously unused sites to the quality assurance agency (QAA).

1.8.2. SNL:

1.8.2.1. Providing, calibrating, and operating instruments and sampling devices, except for those used for SF₆ sampling and analysis.

1.8.2.2. Providing the BB facility including access airlock and mixing fans.

1.8.2.3. Providing CO₂.

1.8.2.4. Providing analyte standards for calibrating real-time instruments.

1.8.2.5. Analyzing results (Tables 1.3 and 1.4).

1.8.3. ELI:

1.8.3.1. QA planning and monitoring of established QC procedures.

1.8.3.2. Arranging for transportation of resin samplers from Alpine West Laboratory (AWL) to SNL.

1.8.3.3. Arranging for transportation of samples from SNL to AWL and Battelle-Columbus Division (BCD).

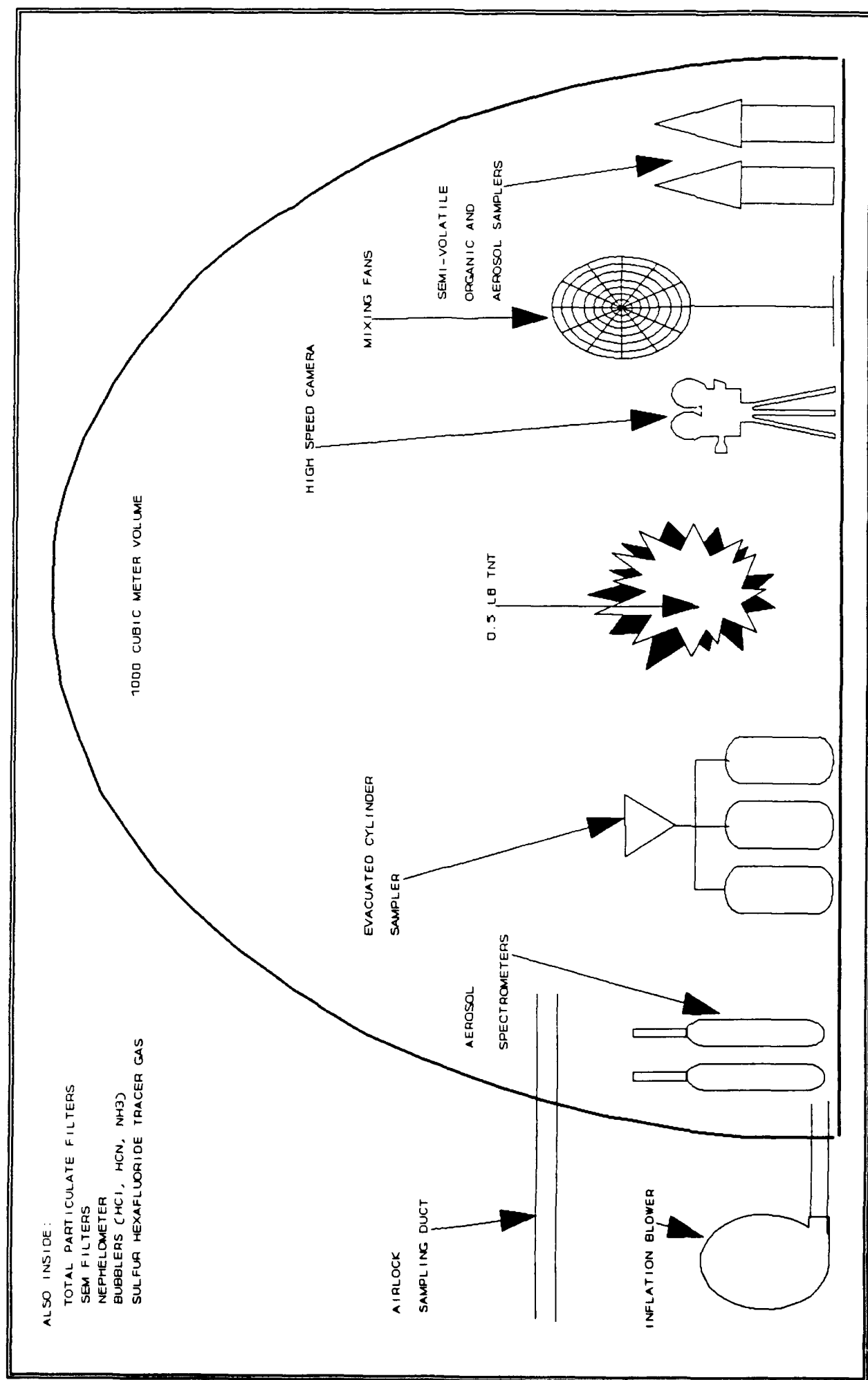


Figure 4.2 SNL BangBox Interior OB/OD Sampling Instrumentation and Equipment.

Table 1.3 Completeness of Effort For Sampling and Analysis During BangBox Test.

Type of Analysis	Reference Table	Chemical Species	Assay Method	Approximate Number Samples	Assay Lab Responsible
Exotic Organics	1.5	15 compounds	SFC-MS ^a	145	AWL ^b
	1.6		GC-MS ^c	53	BCD ^d
Inorganic (metal)	1.4	7	XRF ^e	11/15	SNL ^f
Total Particulate Carbon	1.4	NA ^g	Combustion	24	
Volatiles	1.4	THC ^h , CH ₄ , H ₂ , C ₂ -C ₁₀ , C ₆ H ₆ , CO, CO ₂ .	GC ⁱ	9	OGC ^j
Particle Size	1.4	NA	SEM ^k	15	SNL
Near-real Time	NA	SF ₆	GC/ECD ^l	Continuously	OGC
Real-Time	NA	CO, CO ₂ , NO _x ^m , SO ₂ , O ₃ , THC	Gas and Total Hydrocarbon Analyzers	Continuously	SNL
Bubbler	1.4	HCN, HCL, NH ₃	Wet Chemistry	6 ⁿ	SNL
Particle Size/mass	NA	NA	DMPS ^o , APS ^p , ASASP ^q , FSSP ^r	Continuously (60-sec average)	SNL

^aSupercritical fluid chromatography/mass spectrometer.

^bAlpine West Laboratory.

^cGas chromatograph/mass spectrometer.

^dBattelle Columbus Division.

^eX-ray Fluorescence.

^fSandia National Laboratories.

^gNot applicable.

^hTotal hydrocarbons.

ⁱGas chromatograph.

^jOregon Graduate Center.

^kScanning electron microscopy.

^lElectron capture detector.

^mNitrogen oxides.

ⁿThree for background, three for sample cloud.

^oDifferential mobility particle sizer.

^pAerodynamic particle sizer.

^qActive scattering aerosol spectrometer probe.

^rForward scattering spectrometer probe.

Table 1.4 Sampling and Assay^a (Other than Exotic, Real-Time, and Near-Real Time) Requirements for the Sandia BangBox Test.

Type of Sample	Collection Source	Sample Type	Trial 1			Trial 2			Trial 3			Total Assays (min/max)
			BG ^b	NH ^c	HG ^d	BG	NH	HG	BG	NH	HG	
Indirect Sample (1-m ³ bag)	Teflon™ Filter	Inorganic (metals)	1	1	1	0 or 1	1	1	0 or 1	1	1	7/9
Direct Sample			1	1	NA ^e	0 or 1	1	NA	0 or 1	1	NA	4/6
Indirect Sample (1-m ³ bag)	Quartz Filter	Total Particulate Carbon	1	1	1	1	1	1	1	1	1	9/9
Direct Sample (inside BB)			1	1	NA	1	1	NA	1	1	NA	6/6
Direct Sample (32-L Tanks)			1	1	1	1	1	1	1	1	1	9/9
Direct Sample (6-L Canister)	Air	THC ^f , CH ₄ , H ₂ C ₂ -C ₁₀ , C ₆ H ₆ , CO, CO ₂ .	1	2	2	1	2	2	1	2	2	18/18 ^g
Indirect Sample (6-L Canister)	Air	THC _f , CH ₄ , H ₂ C ₂ -C ₁₀ , C ₆ H ₆ , CO, CO ₂ .	1	1	2	1	1	2	1	1	2	12/12
Indirect Sample (1-m ³ bag)	Nuclepore Filter	Particulate for SEM ^h	1	1	1	1	1	1	1	1	1	9/9
Direct Sample			1	1	NA	1	1	NA	1	1	NA	6/6
Bubbler	Collecting Fluid	HCN, HCL, NH ³	1	1	NA	1	1	NA	1	1	NA	6/6

^aSampling will be accomplished for all collection media on every trial; assay will be performed initially only from some of the collected samples; additional assay will be performed only upon approval of Program Manager.

^bBackground.

^cNon-homogeneous.

^dHomogeneous.

^eNot Applicable.

^fTotal Hydrocarbons.

^gThis includes an additional 3 canister (2 spiked, 1 blank).

^hScanning electron microscopy.

1.8.3.4. Transporting an unspiked "clean" soil sample from DPG to AWL.

1.8.3.5. Shipping 15 aliquots of 250-g homogeneous samples of virgin DPG soil to the U.S. Environmental Protection Agency (EPA) for spiking.

1.8.3.6. Shipping nine samples, 50 g each, of cleaned, virgin XAD-2™ resin and nine samples, 65 g each, of cleaned, virgin Porapak-R™ resin to EPA for spiking.

1.8.4. AWL:

1.8.4.1. Obtaining and providing resin filters, quartz filters, and glass cylinders that support the resin sampling trains.

1.8.4.2. As requested: Dry, homogenize, and prepare for shipment to EPA, 15 aliquots of 250-g virgin DPG soil samples.

1.8.4.3. Analyzing a TNT block sample from the same lot as that used in the explosion trials.

1.8.4.4. Analyzing "exotic" organic compounds (Table 1.5).

1.8.4.5. Developing letters of instruction (LOIs) for AWL analytical procedures.

1.8.4.6. Analyzing mixing fan blade swab samples.

1.8.5. BCD:

1.8.5.1. Analyzing "exotic" organic compounds (Table 1.6).

1.8.5.2. Developing LOIs for BCD analysis procedures.

1.8.6. Oregon Graduate Center (OGC):

^aSampling will be accomplished for all collection media on every trial; assay will be performed initially only from some of the collected samples, additional assay will be performed only upon approval of Program Manager.

^bBackground.

^cNon-homogeneous.

^dHomogeneous.

^eThe quartz filter from VOST 2A and 2B will be used for total particulate carbon analysis. The quartz filter from the 32-L tanks set-up will be cut into sections so that the exotic organics and total particulate carbon can be analyzed.

^fNot applicable.

^gThree 32-L tanks will sample in parallel (1 manifold) at each time period.

Table 1.6 Battelle-Columbus Division Assay^a Requirements for the Exotic Organics in the Sandia BangBox Test.

Type of Sample	Sample Type	Collection Source	Trial 1 BG ^b NH ^c HG ^d	Trial 2 BG NH HG	Trial 3 BG NH HG	Total Assays
Indirect Sample (1-m ³ bag)	VOST ^e 1A	Quartz Filter	1 0 1	1 0 1	1 0 1	6
		XAD-2™	1 0 1	1 0 1	1 0 1	6
		Porapak-R™	0 0 1	0 0 1	0 0 1	3
Indirect Sample (1-m ³ bag)	VOST ^e 2A	Quartz Filter	0 0 0	0 0 0	0 0 0	0
		XAD-2™	0 0 1	0 0 1	0 0 1	3
		Porapak-R™	1 0 1	1 0 1	1 0 1	6
Direct Sample	VOST ^e 1B	Quartz Filter	1 1 NA ^f	0 0 0	0 0 0	2
		XAD-2™	1 1 NA	0 0 0	0 0 0	2
		Porapak-R™	0 1 NA	0 0 0	0 0 0	1
Direct Sample	VOST ^e 2B	Quartz Filter ^e	0 0 NA	0 0 0	0 0 0	0
		XAD-2™	0 1 NA	0 0 0	0 0 0	1
		Porapak-R™	1 1 NA	0 0 0	0 0 0	2
Direct Sample	32_L Tanks ^g	Quartz Filter	0 0 0	0 0 0	0 0 0	0
		Reflux Extract	1 0 1	1 0 1	1 0 1	0
Other Samples	Soil Samples	4 spiked virgin soil samples (two levels of spiking, each in duplicate). 1 virgin soil sample sent as handling control to EPA.				4 1
	Spiked Resin	2 XAD-2™ (1 level of spiking in duplicate) 2 Porapak-R™ (1 level of spiking in duplicate)				2 2
	Handling Control	1-XAD-2™ 1-Porapak-R™				1 1
		32-L Tanks	2 (1 level of spiking in duplicate)			2
Fan Blade		Swap or wash 1 sample at end of each trial (1 blade)				1
Spiking Solution		1 Solution spiked with exotics				1
Total						53

^aSampling will be accomplished for all collection media on every trial; assay will be performed initially only from some of the collected samples; additional assay will be performed only upon approval of Program Manager.

^bBackground.

^cNon-homogeneous.

^dHomogeneous.

^eThe quartz filter from VOST 2A and 2B will be used for total particulate carbon analysis. The quartz filter from the 32-L tanks set-up will be cut into sections so that the exotic organics and total particulate carbon can be analyzed.

^fNot applicable.

^gThree 32-L tanks will sample in parallel (1 manifold) at each time period.

1.8.6.1. SF₆ sampling and analysis by gas chromatograph/electron capture detector (GC/ECD).

1.8.6.2. Providing SF₆ and release mechanism.

1.8.6.3. Providing 6-L SS canisters and 32-L SS tanks.

1.8.6.4. Analyzing 6-L canister contents (THC, CH₄, C₂-C₁₀, C₆H₆, CO, CO₂, H₂).

1.8.6.5. Furnishing three 6-L SS canisters under vacuum to EPA for spiking (2 will be spiked, 1 will be blank).

1.8.6.6. Analyzing the 6-L spiked canisters for THC, CH₄, C₂-C₁₀, C₆H₆, CO, CO₂.

1.8.6.7. Furnishing two 32-L SS tanks under vacuum to EPA for spiking with the same set of exotics as used to spike soils and trapping resins (both will be spiked).

1.8.6.8. Reflux-extracting the contents of the 32-L tanks at SNL or OGC on site and transporting solutions for concentration and analysis by AWL and BCD.

1.8.6.9. Developing LOIs for OGC laboratory analysis procedures.

1.8.6.10. Analyzing results of sampling outlined in Tables 1.3 and 1.4.

1.8.7. U.S. Army Armament, Munitions, and Chemical Command (AMCCOM):

Providing twenty 0.5-lb (227-g) TNT blocks from one lot and 20 blasting caps.

1.8.8. SSL:

1.8.8.1. Aerosol carbon analyses.

1.8.8.2. Developing of LOIs for SSL analysis procedures.

1.8.8.3. Analyzing results of sampling outlined in Tables 1.3 and 1.4.

1.8.9. EPA, Research Triangle Park:

1.8.9.1. Spiking aliquots of 12 virgin soil samples with some of the potential contaminants/combustion product compounds (Table 1.7) dissolved in acetone at two different levels (6 of each). Two of each level of spiking will be sent to AWL and two to BCD. The remaining set of four will be retained by EPA. The homogeneous soil samples, 250 g each, will be shipped in glass sample jars with Teflon™-lined caps. In addition, one unspiked, virgin soil sample will be sent to each AWL and BCD. EPA will retain one unspiked, virgin soil sample. All samples sent to AWL and BCD will be unlabeled as to condition (spiked and unspiked).

1.8.9.2. Spiking six 50-g XAD-2™ and six 65-g Porapak-R™ resin samples, at one level with a selection of compounds between number 10 and 23 in Tables 1.7, dissolved in acetone. These samples will be accompanied by three unspiked 50-g samples of XAD-2™ resin and (three unspiked 65-g samples of Porapak-R™). A set of six of these resins (two XAD-2™ samples spiked at one level plus a blank and two Porapak-R™ samples spiked at one level plus a blank) will be sent to AWL; another like set will be sent to BCD and a third like set will be retained by EPA.

1.8.9.3. Identically spiking two 32-L tanks with a selection of compounds between numbers 10 and 23 in Table 1.7. Tanks will be sent to OGC for reflux-extraction.

1.8.9.4. Identically spiking two 6-L canisters with a selection of compounds from the list furnished by OGC. In addition, one 6-L canister will be blank. These will be sent to OGC for analysis.

1.8.10. Andrulis Research Corporation:

1.8.10.1. Observing the test.

1.8.10.2. Analyzing data.

1.8.10.3. Preparing the test report.

Table 1.7 Analyte Product List and Laboratory Responsible for Analyses.

Number	Analyte	Laboratory Responsible
1	Total hydrocarbon	Sandia National Laboratories
2	Methane, C ² -C ¹⁰ , hydrogen, benzene	Oregon Graduate Center
3	Carbon monoxide	Sandia National Laboratories
4	Carbon dioxide	
5	Sulfur dioxide	
6	Oxides of nitrogen	
7	Ozone	
8	Sulfur Hexafluoride	Oregon Graduate Center
9	Hydrogen cyanide, hydrogen chloride, ammonia	Sandia National Laboratories
10	Trinitrotoluene	Alpine West Laboratories and Battelle-Columbus Division
11	Phenol	
12	4-Nitrophenol	
13	2,4- & 2,6-Dinitrotoluene	
14	N-Nitrosodiphenylamine	
15	Benz[a]anthracene	
16	Benz[a]pyrene	
17	Dibenz[a,h]anthracene	
18	Benz[c]acridine	
19	Dibenzofuran	
20	1-Naphthalenamine	
21	2-Nitronaphthalene	
22	1-Nitropyrene	
23	1,6-Dinitropyrene	
24	Antimony	Lawrence-Berkeley Laboratories
25	Barium	
26	Chromium	
27	Lead	
28	Nickel	
29	Cadmium	
30	Mercury	

1.9. Safety

The only explosively hazardous material used during this test will be TNT and blasting caps. Existing, approved SNL safety procedures for the handling, storage, preparation, and detonation of these items within the test facility will be followed.

1.10. National Environmental Policy Act (NEPA) Process

The potential environmental impacts of the proposed test program will be reviewed in light of the requirements of the NEPA and applicable Department of Energy and Department of the Army implementing instructions. Prior to initiating BB testing, the results of NEPA review will be documented and approved by the appropriate element at SNL.

1.11. Quality Assurance

A QA protocol and QC procedures will be prepared by the QAA prior to the beginning of the test which will, after approval by the Program Manager (PM), be returned to the QAA before initiation of testing.

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SECTION 2. SUBTESTS

2.1. CO₂ LEAK RATE DETERMINATION

2.1.1. Objective

The objective of this subtest is to determine the BB air leak rate under normal operating conditions with the BB inflation blower on and also with the blower off.

2.1.2. Data Required

2.1.2.1. The volume, at ambient temperature and pressure, of CO₂ released into the BB chamber (± 2 percent);

2.1.2.2. BB chamber volume (± 2 percent);

2.1.2.3. CO₂ concentration (weight/unit volume and ppm), continuously monitored from time of event (T)-5 min to end of trial (in this instance, "T" is time of release of CO₂ into the BB chamber) (± 2 ppm);

2.1.2.4. Ambient atmospheric pressure (± 2 mb);

2.1.2.5. Pressure differential between the BB interior and ambient atmosphere as continuously monitored from T-5 min to the end of trial (± 2 mb);

2.1.2.6. Time (s) required to fill a 1-m³ air bag (relative to "T");

2.1.2.7. Facility interior temperature ($\pm 0.5^{\circ}\text{C}$);

2.1.2.8. Ambient exterior temperature ($\pm 0.5^{\circ}\text{C}$); and,

2.1.2.9. Dew point of the BB atmosphere ($\pm 0.5^{\circ}\text{C}$).

2.1.3. Data Acquisition Procedures

2.1.3.1. Mixing fans will operate for a period prior to T to eliminate or minimize the vertical temperature differential within the facility. At T+0 min, a quasi-instantaneous release of CO₂ will occur; the CO₂ will be allowed to mix and diffuse throughout the facility. The period for monitoring the CO₂ cloud will be from T-5 min to at least T+35 min, but will not exceed T+60 min unless otherwise determined by the PM. For the trial with the inflation blower operating, the CO₂ concentration will continue to be monitored until ambient levels are achieved. A minimum of two trials will be run. One trial will be conducted with the inflation blower operating continuously and a second trial with the inflation blower turned off after the CO₂ dissemination.

2.1.3.2. Any modification to the facility after the two initial CO₂ trials may necessitate conduct of additional CO₂ trials.

2.1.4. Analytical Procedures

2.1.4.1. The real-time CO₂ concentration data will be examined over the sampling period to determine the inflation blower condition and the mixing fan condition (on or off) that will be used in the next series of trials (SF₆ and TNT). The examination of the CO₂ real time concentration data will be accomplished by the OB/OD consultant team using the expected concentration based on the amount of CO₂ disseminated and the facility volume.

2.1.4.2. The time (s) required to fill the 1-m³ air bag will be measured.

2.2. HOMOGENEITY

2.2.1. Objectives

The objective of this subtest are:

2.2.1.1. To establish the mixing time after detonation needed to attain homogeneity of the TNT detonation cloud within the BB.

2.2.1.2. To ensure that all real-time systems are operational.

2.2.1.3. To ensure that test design (placement of sampling equipment, sampling probes, TNT, SF₆ and photo/video equipment) is operationally satisfactory for the full-scale sampling trials.

2.2.2. Data Required

2.2.2.1. The volume/weight, at ambient temperature and pressure, of SF₆ released into the BB (± 2 percent).

2.2.2.2. BB volume (± 2 percent) [determined by calculation from SF₆ (when homogeneity is established) and by measurement of internal dimensions].

2.2.2.3. Analog data (60-s averages) from T-5 to T+35 min (or longer) for the following instruments:

2.2.2.3.1 Nephelometer.

2.2.2.3.2 CO₂ analyzer (± 2 ppm).

2.2.2.3.3 CO analyzer (± 0.05 ppm).

2.2.2.3.4 NO_x analyzer (± 5 ppb).

2.2.2.3.5 SO₂ analyzer (± 5 ppm).

2.2.2.3.6 O₃ analyzer (± 2 ppb).

2.2.2.3.7 PID (± 1 ppm).

2.2.2.3.8 DMPS.

2.2.2.3.9 APS.

2.2.2.3.10 ASASP.

2.2.2.3.11 FSSP

2.2.2.3.12 Pressure (ambient, exterior) (± 2 mb) not in real-time [just read gauge].

2.2.2.3.13 Test chamber interior-ambient differential pressure (± 2 mb) not in real time [just read gauge].

2.2.2.3.14 Test chamber temperature ($\pm 0.5^\circ\text{C}$).

2.2.2.3.15 Test chamber dew/frost point ($\pm 0.5^\circ\text{C}$).

2.2.2.3.16 Bag sample valve position.

2.2.2.3.17 Gas analyzer valve position.

2.2.3. Data Acquisition Procedures

Mixing fans will operate for 5 min prior to T to eliminate or minimize the vertical temperature differential within the BB. At T-time, the 227 g of TNT will be detonated simultaneously with release of a known quantity of SF_6 . An 0.85-L canister of SF_6 will be located 4.6 m from the charge and connected to the high-speed camera for release. The release point will be approximately 25 cm above the floor. The TNT block will be suspended 1 m above ground level. Mixing fans will be sequenced (on and off) according to a plan developed for the CO_2 trials (Paragraph 2.1.3above). All real-time and near-real-time monitors of the aircraft sampling system will be recorded from T-5 to T+35 min, or longer.

2.2.4. Analytical Procedures

The real-time CO₂ and near-real-time SF₆ (digital output and graphic display) data will be examined over the sampling period to determine onset of homogeneity within the BB during an actual detonation of TNT. Other real-time data will also be examined for the background levels before detonation and for the post-detonation levels of the gases, as well as for the particulate distribution within the aerosol cloud. The data will then be examined by the on-site consultant team. Recommendations will be made to the PM on the sequence of events for the subsequent series of trials to be conducted with the full array of sampling.

2.3. EXTENDED BACKGROUND SAMPLING

2.3.1. Objective

To determine, by extended sampling, background levels of semivolatile and non-volatile organic compounds in the atmosphere adjacent to the BB, and inside the BB chamber.

2.3.2. Data Required

2.3.2.1. SFC/MS analysis of VOST cartridge resins.

2.3.2.2. GC/MS analysis of VOST cartridge resins.

2.3.3. Data Acquisition Procedures

2.3.3.1. Two high-volume direct sampling systems (100-L/min capacity) will each be configured with a sampling train consisting of one cartridge containing 65 g of XAD-2™ resin, and a second cartridge containing 20 g of XAD-2™ resin. These cartridges will be in series, the 65-g cartridge being the lead cartridge.

2.3.3.2. Sampling systems will be continuously operated adjacent to the BB building for 6 hr, then moved inside the BB chamber and restarted with fresh resin cartridges and operated continuously for an additional 6 hr.

2.3.3.3. The operation, handling, and assay of the samples are described in LOIs specifically written for this test.

2.3.3.4. Sampler flow-rate readings will be taken once every 2 hr.

2.3.4. Analytical Procedures

2.3.4.1. Samples will be assayed using SFC/MS and GC/MS. Analytical procedures are contained in separate LOIs.

2.3.4.2. QA/QC will be conducted and maintained throughout the entire test process. The QA director, or his representative, will selectively monitor pretrial and trial activities, conduct of trials and sample/data collection, extraction, analysis, and data recording (consistent with the QA/QC plan) as approved by the PM. Observations will include adherence to LOIs, laboratory procedures, and preparation of data for subsequent analysis. The QAA is responsible for shipping specimens and for archiving specimens and original data hard-copy printouts and/or computer diskettes. Individual laboratories are responsible for QC in their laboratories.

2.4. TRINITROTOLUENE SINGLE DETONATION

2.4.1. Objectives

2.4.1.1. To provide a cross-check on the calculations of the expected mass of combustion products between the carbon balance technique and the cloud volume/cloud concentration technique, under controlled conditions.

2.4.1.2. To verify the validity of measurement and sampling techniques proposed for use on the FWAC (on subsequent OB/OD field tests) by comparison of the analysis results with those from other methods, especially bag sampling vs. evacuated tank sampling.

2.4.1.3. To establish the technical suitability of supercritical fluid chromatography/mass spectrometry (SFC/MS) for analysis of both aerosol and gas-phase organic samples from OB/OD trials as compared to the more conventional gas chromatography/mass spectrometry (GC/MS) analysis methods.

2.4.1.4. To provide preliminary information (for planning purposes) on the morphology, composition, and size distribution of airborne particulate material and gases/vapors generated by high explosive (TNT) detonations.

2.4.2. Data Required

2.4.2.1. Video coverage from T-1 to T+35 min by a camera inside the test chamber and by an outside camera showing the BB as a whole.

2.4.2.2. Motion picture coverage at 5,000 frames/s from T+0 to T+2 s inside the chamber. A wide-angle lens will be used to document width of the fireball.

2.4.2.3. One PMS probe (both ASASP 100X and FSSP 100X) particle size distributions determinations/min, from T-45 to T+35 min.

2.4.2.4. One APS particle size distribution determination/min, from T-45 to T+35 min.

2.4.2.5. One DMPS particle size distribution/5 min, from T-45 to T+35 min.

2.4.2.6. Analog data (60-s averages), from T-45 to T+35 min from the following instruments:

2.4.2.6.1 Nephelometer.

2.4.2.6.2 CO₂ analyzer (± 2 ppm).

2.4.2.6.3 CO analyzer (± 0.5 ppm).

2.4.2.6.4 NO_x analyzer (± 5 ppb).

2.4.2.6.5 SO₂ analyzer (± 5 ppm).

2.4.2.6.6 O₃ analyzer (± 2 ppb).

2.4.2.6.7 PID for organic analyses (± 1 ppm).

2.4.2.6.8 Ambient pressure (exterior) (± 2 mb).

2.4.2.6.9 BB chamber interior/exterior (ambient) differential pressure (± 2 mb).

2.4.2.6.10 BB chamber temperature ($\pm 0.5^\circ\text{C}$).

2.4.2.6.11 BB chamber dew/frost point ($\pm 0.5^\circ\text{C}$).

2.4.2.6.12 Bag sampler valve position.

2.4.2.6.13 Gas analyzer valve position.

2.4.2.7. Analog data (60-s average) from the following instruments on bag samples taken at T-30, T+3, and T+15 min (or later, if need be, to ensure the homogeneity of the detonation products within the BB chamber) for the period during which the air bag is being pumped down:

2.4.2.7.1 CO₂ analyzer (± 2 ppm).

2.4.2.7.2 CO analyzer (± 0.5 ppm).

2.4.2.8. SFC/MS analysis of each component (filters and resins) of the VOST samplers operated inside the test chamber for volatile, semivolatile, and nonvolatile organics (Table 1.5).

2.4.2.9. SFC/MS analysis of each component (filters and resins) of the VOST samplers sampled from the 1-m³ air bag (Table 1.5).

2.4.2.10. GC/MS analysis as designated in Table 1.6 of each component (filters and resins) of the VOST samplers operated inside the test chamber for volatile, semivolatile, and nonvolatile organics.

2.4.2.11. GC/MS analysis designated in Table 1.6 of each component (filters and resins) of the VOST samples collected from the 1-m³ air bag.

2.4.2.12. Analyses for THC, CH₄, C₂-C₁₀, C₆H₆, CO, CO₂, and H₂ contained in 6-L sampling canisters operated at T-30, T+3, T+15, and T+30 min.

2.4.2.13. X-ray fluorescence (XRF) elemental analysis of Teflon™ filters exposed inside the BB chamber from T-45 to T-15 min and from T+2 to T+35 min.

2.4.2.14. XRF elemental analysis of Teflon™ filter samples exposed to the air drawn from the bag samples obtained at T-30, T+3, T+15, and T+30 min.

2.4.2.15. SEM/X-ray energy dispersive analysis (XEDA) of particulate from Nuclepore™ filters exposed within the test chamber from T-45 to T-15 min and from T+2 to T+35 min.

2.4.2.16. SEM/XEDA analysis of Nuclepore™ filters exposed to the air drawn from the bag samples obtained at T-30, T+3, and T+15 min.

2.4.2.17. Analyses for HCN and NH₃ in bubbler samples obtained from T-45 to T-15 min and from T+2 to T+35 min.

2.4.2.18. Combustion weight loss analysis of quartz filter samples exposed within the test chamber from T-45 to T-15 min and from T+3 to T+35 min.

2.4.2.19. Combustion weight loss analysis of quartz filter sample exposed to the air drawn from the bag samples obtained at T-30, T+3, and T+15 min.

2.4.2.20. Combustion weight loss analysis of quartz filters sample (32-L tank system) exposed within the test chamber at T-30, T+3, and T+15 min.

2.4.2.21. GC/ECD analysis for SF₆ performed as often as possible during the T-45 to T+35 min period of the trial.

2.4.2.22. The instruments listed in paragraphs 1.6.2 and 1.6.3.1 through 1.6.3.5 will sample directly from the BB chamber through a probe when not sampling from the 1-m³ bag.

2.4.2.23. Combustion weight loss analysis of quartz fiber filters.

2.4.3. Data Acquisition Procedures

Based upon review of the real-time and near-real-time data from the CO₂ and the SF₆ trials, respectively, the operating conditions of the BB during the TNT detonation trials and the sampling times for these trials will be determined.

2.4.3.1. Three separate trials will be conducted, each involving the detonation of one 227-g block of TNT.

2.4.3.2. The real-time and near-real-time samplers will be operational from start of background to the end of trial. These sampling results from the FWAC system will be collected on data loggers and then reduced to engineering units by SNL (all raw voltages will be saved for quality check of algorithms and for the archival files.)

2.4.3.3. The grab bag samples (32-L tanks, 6-L canisters, and 1-m³ Velostat™ bag) will be taken at times determined from review of the results from the CO₂ and SF₆ trials. The samples will be from at least three periods during a trial. The periods are: (1) background (prior to TNT

detonation), (2) nonhomogeneity of TNT detonation product aerosol, and (3) once homogeneity of TNT-detonation product aerosol has been achieved.

2.4.3.3.1 The 32-L tank system will consist of a 20- x 25-cm quartz fiber filter followed by a manifold of three 32-L tanks. The three evacuated 32-L tanks will function simultaneously, thus providing a 96-L sample of aerosol at each sampling time. The organic detonation products from the aerosol will then be extracted by reflux-extraction of the interior of the tanks with methylene chloride. The procedures used to extract these tanks will be provided by the OGC representatives prior to conduct of the preliminary trials.

2.4.3.3.2 The 6-L canister system will be used to obtain paired samples directly from the BB and one from the 1-m³ Velostat™ bag. The sample will be withdrawn and analyzed according to LOI procedures to be finalized before conduct of the preliminary trials.

2.4.3.3.3 The 1-m³ Velostat™ bag system (indirect sampling system) will collect the aerosol through a 10-cm diameter aluminum sampling probe extending into the BB (Figure 1.2). The bag will be filled and then the collected aerosol will be pumped through two VOST samplers (Figure 1.1), one Teflon™ filter, one SEM filter, and real-time monitors for CO₂ and CO. LOI procedures for operating this system will be provided prior to conduct of preliminary trials.

2.4.3.4. A high volume direct sampling system (100-L/min capacity) will be located within the BB and consist of two VOST trains, one Teflon™ filter, and one SEM filter (Figures 1.1 and 1.3). This sampling system will be used to sample a large volume of the aerosol and will obtain a background sample from each sampler train and a sample after SF₆ release and TNT detonation. The operation, handling, and assay of the samples will be described in an LOI to be provided prior to conduct of preliminary trials.

2.4.3.5. Particle sizing of the aerosol will be accomplished with the ASASP aerosol probe and the FSSP aerosol probe located within the BB and with the DMPS and APS sampling systems through a probe extending into the BB. The operation and data output will be provided as an LOI prior to conduct of preliminary trials.

2.4.3.6. The operation of the video and high-speed cameras will be outlined in an LOI to be provided prior to conduct of preliminary trials.

2.4.4. Analytical Procedures

2.4.4.1. Sample analysis procedures for permanent gas and volatile organic species (by GC), for other organic species (by SFC/MS and GC/MS), for elemental content (by XRF), for carbon (by pyrolysis/combustion techniques), and for TSP (by gravimetric analysis) will be specified in separate LOIs to be appended to the test plan.

2.4.4.2. The results of the sample analyses, together with the real-time and near-real-time data, will be interpreted by the consultant team using both the cloud volume/cloud concentration model or method (taking into account that the cloud volume is fixed by the volume of the chamber), and by the carbon balance model. The comparison of these results will indicate whether, on subsequent field tests, the simpler carbon balance method can be used exclusively, the cloud volume/cloud concentrations model can be used exclusively, or a combination of both should be used.

2.4.4.3. The consultant team will also review all other data that is developed on-site to evaluate whether the measurement and sampling techniques used during this test (and proposed for subsequent use on the DPG FWAC tests) are adequate and whether the SFC/MS analysis method can be used on future field trials as the principal separation and analysis technique for determining the concentrations and identities of organic emissions from OB/OD tests.

2.4.4.4. QA/QC. Paragraph 2.3.4.2 applies.

2.5. Trinitrotoluene Multiple Detonation

2.5.1. Objectives

2.5.1.1. To collect, using XAD-2™ filters, a sufficient sample of detonation emissions to facilitate detection of very low levels of trace/exotic organic detonation products.

2.5.1.2. To establish the technical suitability of SFC/MS for analysis of both aerosol and gas-phase organic samples from OB/OD trials as compared to the more conventional GC/MS analysis methods.

2.5.1.3. To provide preliminary information (for planning purposes) on the morphology, composition, and size distribution of airborne particulate material and aerosols/vapors generated by high explosive (TNT) detonations.

2.5.2. Data Required

2.5.2.1. Video coverage. Paragraph 2.4.2.1 applies.

2.5.2.2. Particle size distribution. Paragraphs 2.4.2.3- 2.4.2.5 apply.

2.5.2.3. Analog data. Paragraph 2.4.2.6 applies. Real-time and near-real-time data acquisition may be reduced to once every 15 s.

2.5.2.4. SFC/MS analysis. Paragraph 2.4.2.8 applies.

2.5.2.5. GC/MS analysis. Paragraph 2.4.2.10 applies.

2.5.2.6. Bubbler solvent analysis for HCN and NH₃. Paragraph 2.4.2.17 applies.

2.5.2.7. VOST flow rates as measured between detonations events.

2.5.3. Data Acquisition Procedures

2.5.3.1. Eight 227-g TNT blocks will be sequentially detonated, one per hr, during a 7-hr period. Each explosive charge will be prepared for detonation, and detonated, in a manner identical to that used in the single-detonation subtest.

2.5.3.2. Real-time and near-real-time samplers. Paragraph 2.4.3.2 applies.

2.5.3.3. Direct-sampling system. A high-volume direct sampling system (100-L/min capacity) will be located within the BB and consist of two VOST trains, each equipped with quartz filters (Figures 1.1 and 1.3).

2.5.3.3.1 The system will be operated continuously from T+2 until T+47 following each detonation.

2.5.3.3.2 Resin filters will not be changed during the subtest.

2.5.3.3.3 Quartz fiber filters will be changed after the fourth detonation.

2.5.3.4. Particle size distribution. Paragraph 2.4.3.5 applies.

2.5.3.5. Video recording. An LOI describing operation of video cameras will be prepared.

2.5.4. Analytical Procedures

2.5.4.1. Sample analysis procedures for permanent gas and volatile organic species (by GC) for other organic species (by SFC/MS and GC/MS) are specified in separate LOIs.

2.5.4.2. Carbon balance method validation. Paragraph 2.4.4.2 applies.

2.5.4.3. Application to FWAC field test sampling. Paragraph 2.4.4.3 applies.

2.5.4.4. QA/QC. Paragraph 2.3.4.2 applies.

2.6. Trinitrotoluene Multiple Tank Sampling

2.6.1. Objectives

2.6.1.1. To collect, in 32-L tanks and quartz filters, sufficient detonation emissions to facilitate detection of very low levels of trace/exotic organic detonation products free from the potential matrix effects of resins.

2.6.1.2. To establish the technical suitability of SFC/MS for analysis of both aerosol and gas-phase organic samples from OB/OD trials as compared to the more conventional GC/MS analysis methods.

2.6.1.3. To provide preliminary information (for planning purposes) on the morphology, composition, and size distribution of airborne particulate material and aerosols/vapors generated by high explosive (TNT) detonations.

2.6.2. Data Required

2.6.2.1. Video coverage. Paragraph 2.4.2.1 applies.

2.6.2.2. Particle size distribution. Paragraphs 2.4.2.3- .5 or 2.4.2.3- 2.4.2.5 apply.

2.6.2.3. Analog data. Paragraph 2.4.2.6 applies.

2.6.2.4. SFC/MS and GC/MS assay. Half of the extract will be designated for SFC/MS assay and the other half designated for GC/MS assay.

2.6.2.5. Combustion weight loss analysis. Quartz filter samples will be subjected to combustion weight loss analysis.

2.6.3. Data Acquisition Procedures

2.6.3.1. Real-time and near-real-time samplers. Paragraph 2.4.3.2 applies.

2.6.3.2. Tank sampling. Nine 32-L tank systems will each consist of a 20- x 25-cm quartz fiber filter followed by a manifold of three 32-L tanks. Three tanks will be set in Dewar flasks containing liquid nitrogen so that the sample will be condensed and the equivalent of 10 atmospheres of sample captured. The 27 evacuated 32-L tanks will function simultaneously at T+5. The organic detonation products from the aerosol will then be extracted from all tanks by reflux-extraction using methylene chloride. The procedure used to extract these tanks is in described LOIs.

2.6.3.3. Particle size distribution. Paragraph 2.4.2.5 applies.

2.6.3.4. Video recording. An LOI describing operation of video recorders will be prepared.

2.6.4. Analytical Procedures

Paragraph 2.3.4 applies.

2.7. Double-base Propellant Burn

2.7.1. Objectives

2.7.1.1. To verify the validity of measurement and sampling techniques proposed for use on the FWAC (on subsequent OB/OD field tests).

2.7.1.2. To provide preliminary information (for planning purposes) on the morphology, composition, and size distribution of airborne particulate material and aerosols/vapors generated by propellant burns.

2.7.2. Data Required

2.7.2.1. Video coverage, including high-speed video, from T-1 to T+35 min by a camera inside the test chamber.

2.7.2.2. Particle size distribution. Paragraphs 2.4.2.3- .5 apply.

2.7.2.3. Analog data. Paragraph 2.4.2.6 applies.

2.7.2.4. SFC/MS analysis. Paragraph 2.4.2.8 applies.

2.7.2.5. GC/MS analysis. Paragraph 2.4.2.10 applies.

2.7.2.6. XRF elemental analysis. Paragraph 2.4.2.13 applies.

2.7.2.7. SEM/XEDA analysis. Paragraph 2.4.2.15 applies.

2.7.2.8. Bubbler solvent analysis for HCN and NH_3 . Paragraph 2.4.2.17 applies.

2.7.2.9. VOST flow rate as measured before and after the burn trial.

2.7.3. Data Acquisition Procedures

2.7.3.1. A stainless steel bowl containing 1 lb (454 g) of NOSIH-AA2 double-based propellant (Table 1.1) and 26.6 g of ethyl cellulose wrapping material will be placed within the BB chamber and ignited with an electric match.

2.7.3.2. Real-time and near-real-time samplers. Paragraph 2.4.3.2 applies.

2.7.3.3. Direct sampling system. A high-volume direct sampling system (100-L/min capacity) will be located within the BB and consist of two VOST trains, one Teflon™ filter, and one SEM filter (Figures 1.1 and 1.3). This sampling system will be used to sample a large volume of the aerosol and will obtain a background sample from each sampler train. The operation, handling, and assay of the samples are described in a separate LOI.

2.7.3.4. Particle size distribution. Paragraph 2.4.3.5 applies.

2.7.3.5. The operation of the video, including high-speed video, is described in a separate LOI.

2.7.4. Analytical Procedures

Paragraph 2.4.4 applies.

2.8. Composite Propellant Burn

2.8.1. Objectives

Paragraph 2.7.1 applies

2.8.2. Data Required

2.8.2.1. Video coverage, including high-speed video, from T-1 to T+35 min by a camera inside the test chamber.

2.8.2.2. Particle size distribution. Paragraphs 2.4.2.3- .5 apply.

2.8.2.3. Analog data. Paragraph 2.4.2.6 applies.

2.8.2.4. SFC/MS analysis. Paragraph 2.4.2.8 applies.

2.8.2.5. GC/MS analysis. Paragraph 2.4.2.10 applies.

2.8.2.6. XRF elemental analysis. Paragraph 2.4.2.13 applies.

2.8.2.7. SEM/XEDA analysis. Paragraph 2.4.2.15 applies.

2.8.2.8. Analysis for Hcl, HCN and NH_3 in bubbler solvent samples obtained from T-45 to T-15 min and from T+2 to T+35 min.

2.8.2.9. Dioxin level analysis.

2.8.2.10. VOST flow rate as measured before and after the burn trial.

2.8.3. Data Acquisition Procedures

2.8.3.1. A stainless steel bowl containing 1 lb (454 g) of Mk 6 Product Improvement Program Mix 217 propellant (Table 1.2) will be placed within the BB chamber and ignited with an electric match.

2.8.3.2. Real-time and near-real-time samplers

Paragraph 2.4.3.2 applies.

2.8.3.3. Direct sampling system. A high-volume direct sampling system (100-L/min capacity) will be located within the BB and consist of two VOST trains, one Teflon™ filter, and one SEM filter (Figures 1.1 and 1.3). This sampling system will be used to sample a large volume of the aerosol so as to obtain a background sample from each sampler train. The operation, handling, and assay of the samples are described in a LOI written specifically for this test.

2.8.3.4. Particle size distribution. Paragraph 2.4.3.5 applies.

2.8.3.5. Video documentation. The operation of video recorders, including high-speed video.

2.8.3.6. Dioxin sampling. Two sampling trains, each consisting of one quartz fiber filter and one polyurethane foam filter, will be operated to obtain a 2-hr background sample. Replacement filters will be inserted prior to composite propellant burns and will be operated from T-2 to T+120 min.

2.8.4. Analytical Procedures

Paragraph 2.4.4 applies.

APPENDIX B. LETTERS OF INSTRUCTION

During BangBox testing, letters of instruction (LOIs) were modified and revised to incorporate lessons learned. In some instances, no changes were necessary; in others, considerable revision occurred before testing concluded. The LOI's in this appendix reflect procedures used as the test ended and which were expected to be used during future tests.

<u>LOI</u> <u>NUMBER</u>	<u>TITLE</u>	<u>PAGE</u>
LOI 1	General Laboratory Procedures	B-3
LOI 2	Preparation, Handling, and Extraction of Quartz Fiber Filters	B-7
LOI 3	Analysis of Bulk Explosives and Propellants	B-9
LOI 4	Soxhlet Extractor Operation	B-11
LOI 5	Rotary Evaporator Operation	B-15
LOI 6	Supercritical Fluid Chromatography/Mass Spectrometry	B-17
LOI 7	Quality Control Plan: Procedures for Accuracy, Precision, and Completeness.	B-25
LOI 8	Preparation and Handling of 32-l Tank Extracts	B-29
LOI 9	Analysis of the Extract from the Spiked 32-l Tanks	B-31
LOI 10	Analysis of the 32-L Tank Extracts	B-33

LOI 11	Preparation and Handling of XAD-2™ Resin Traps, Filters, and VOST	B-35
LOI 12	Sample Preparation for GC/MS and Storage of OB/OD Samples	B-41
LOI 13	Gas Chromatography/Mass Spectrometry (GC/MS) Analysis of OB/OD Samples	B-45
LOI 14	Explosive Preparation for OB/OD BangBox Trials	B-53
LOI 15	Trinitrotoluene (TNT) Detonation	B-57
LOI 16	Data Reduction and Analysis	B-75
LOI 17	VOC Collection Analysis System	B-79
LOI 18	Procedures for Writing Letters of Instruction (LOI)	B-99
LOI 19	LOI for Metals Analysis by X-Ray Fluorescence Analysis	B-103

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LOI 1 GENERAL LABORATORY PROCEDURES

A. Preparation and Cleaning of Glass Sample Storage Containers

Glass containers are washed with hot tap water and detergent, rinsed with distilled water, placed in a concentrated nitric acid bath for 10 min, rinsed again with distilled water, placed in a 10% KOH/isopropanol base bath for 10 min, rinsed again with distilled water, and finally dried in an oven at 110°C for > 8h. The lids are lined with Teflon™ to prevent contamination of the sample.

B. Handling of Filters and Soils

Clean cotton (100%) gloves are used whenever handling soils or filters. In limited situations, Latex gloves may be used.

C. Labeling of Sample Storage Containers

Labels containing the date of use, ELI sample number, and other sample identification information are placed on glass storage containers immediately after sampling and return of the samples (e.g., soil samples, filters, etc.) to the storage containers. Likewise, similar information is recorded in a journal. Both the label and the journal record are signed and dated by AWL authorized personnel.

D. Storage of Filters, Soils, and Fall-out Pan Particulates

All soil and fall-out pan particulate samples are stored in acid-washed glass containers in a walk-in freezer (WIDB 629) at 5°C prior to extraction. All filter samples are stored in their Teflon™-lined envelopes in a freezer at -20°C prior to extraction.

E. Storage of Acetonitrile Extracts

Glass vials (1-dram amber with Teflon™-lined lids) containing the final extracts are stored at -20°C in a freezer.

F. Recording and Correcting Data

1. All data are recorded using a black permanent ink pen.
2. All dates are written in the order of day, month, year, the month being a three-letter abbreviation (i.e., 25 Nov 1988).
3. All times are stated according to a twenty-four hour clock with a colon separating the hour from the minute (i.e., 13:52).
4. All data sheets are signed and dated when completed and only after a check for completeness and correctness has been done.
5. All journals are signed and dated at end of each day and at least on each page.
6. A correction consists of the incorrect data crossed out with a single line such that it remains legible, the correct data written in, date of correction, and initials of person correcting.

7. Any reprocessing is considered new data and not a correction, and is handled as such.

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Study Director	Quality Assurance Director
_____	_____
Date	Date

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LOI 2 PREPARATION, HANDLING, AND EXTRACTION OF QUARTZ FIBER FILTERS

A. Preparation of Filters

1. Quartz fiber filters (5.5-cm round, 10.16-cm round, 20.3-cm x 25.4-cm rectangular) are purchased from Whatman. Cotton (100%) gloves are used for handling the filters. In certain situations, Latex gloves may be used.
2. The filters are placed on aluminum trays, fired in a muffle furnace at 650°C for 8 h, slowly cooled to room temperature, and individually stored in Teflon™-lined envelopes and marked with an I.D. number and weight.

B. Weighing of Filters

1. Twelve hours before weighing, the fired filters are conditioned to the temperature and humidity of the weighing room.
2. The filters are weighed three times with a twelve hour period between each weighing.
3. The filters are weighed both before and after sampling on an analytical balance to 0.01 ng.
4. The filters are placed back in the Teflon™-lined envelopes and labeled with the weight.

C. Labeling of Filters

After sampling, the filters are returned to their labeled storage containers and given another label containing the date of use, ELI sample number, and other identification information. This information is also duplicated in the journal. The label and journal are signed by authorized AWL personnel.

D. Extraction of Filters After Sampling

1. Each filter is placed in a 60-mm X 180-mm cellulose thimble and extracted with 500 mL of nanograde acetonitrile in a Soxhlet extractor (extra large, 1000-mL round bottom flask) for 6 h at a solvent temperature of 80°C.
2. The acetonitrile extract is concentrated to a volume just under 1 mL using a rotary evaporator (50°C), transferred to a 1-dram amber vial (Teflon™-lined cap), and brought to a volume of 1 mL by adding nanograde acetonitrile.
3. The samples are split using a glass micro pipette into two equal 0.5-mL parts, and 100 µL of the internal standard solution is added to one half (300 pg of 1-nitronaphthalene-d₇ and 240 pg of 9-phenylanthracene).

E. Storage of Acetonitrile Extracts

The 1-dram amber vials containing the final filter extracts are stored at -20°C in a freezer.

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LOI 3 ANALYSIS OF BULK EXPLOSIVES AND PROPELLANTS

A. TNT Detonation Blocks

A small sample (<1 g) of TNT from each TNT detonation block is collected and stored in a clean 1-dram glass bottle (Teflon™-lined lid) until analyzed. Approximately 0.1 mg of the TNT is dissolved in 5 mL of methylene chloride for analysis by SFC/MS and GC/MS. A sample (1-mL) of this solution is removed and split into two equal parts. To one half of this sample an internal standard is added (300 pg of 1-nitronaphthalene-d₇ and 240 pg of 9-phenylanthracene). Both halves are placed in storage at -20°C in a freezer.

B. Propellant Samples

Small samples of the double base and composite propellants are collected and stored at -20°C. The samples are treated as in part A.

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LOI 4 SOXHLET EXTRACTOR OPERATION

A. The Soxhlet Extractor consists of three parts: extraction flask, Soxhlet, and condenser (Figure 1).

B. A 1000-mL (extra large) Soxhlet extractor is used with a 60-mm x 180-mm cellulose thimble to extract samples as follows:

1. Cleaning the Extraction Thimble

- a. 500 mL of solvent is placed in the 1000-mL extraction flask.
- b. The extraction flask is placed in the heating mantle as shown in Figure 1.
- c. The Soxhlet extractor is assembled with the cellulose thimble in place.
- d. The cold water to the condenser is turned on.
- e. A variable power transformer is connected to the heating mantle to control the temperature.
- f. The power to the heating mantle is set to a temperature at which the solvent just boils, and there is a turn-over time for the Soxhlet of approximately 10-12 mins.
- g. After completing the required extraction time (approximately 2 h), the power is switched off the heating mantle and the extractor is allowed to cool for 1/2 h.
- h. After cooling, the thimbles are drained of any residual solvent and all solvents are discarded in a waste container.

2. Extracting the Sample

- a. Place the sample in the pre-cleaned thimble.
- b. Follow 1.a-f.

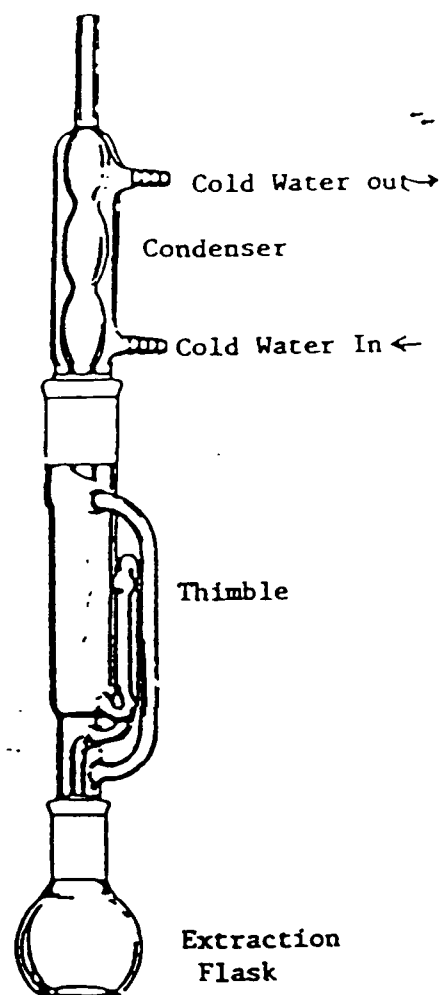


Figure 1. Soxhlet Extractor Set-up

c. After completing the required time, the power is switched off, and the extractor is allowed to cool.

d. After cooling, the thimbles are drained of any residual solvent which is added to the solvent in the extraction flask.

e. The sample is now ready for concentration.

C. After extraction, all Soxhlet parts are cleaned as per LOI-1.

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LOI 5 ROTARY EVAPORATOR OPERATION

1. Turn on the water for vacuum cooling.
2. Turn the vacuum stopcock so that no vacuum is created (aligned with the hole in the condenser).
3. Attach the round bottom evaporating flask containing the sample to the exposed end of the vapor duct with the clip.
4. Lower the evaporating flask so that it is partially submerged in the water bath which should be maintained between 50° and 55°C.
5. Adjust the rotation speed of the drive unit and add the vacuum by rotating the vacuum stopcock one quarter turn. Be certain that no bumping or foaming occurs.
6. When the sample has nearly evaporated (1 mL left), turn off the vacuum and the drive unit. Remove the evaporating flask. Empty the receiving flask in the appropriate waste container.
7. Transfer the remaining solution to a 1-dram glass vial, rinse the round bottom flask with approximately 0.5 mL of solvent 3 times and transfer each rinse to the 1-dram vial.
8. Clean the exposed portion of the vapor duct by the following procedure:
 - a. Turn the vacuum off and turn on the drive unit to a slow setting.
 - b. Hold a 100-mL beaker of appropriate solvent under the rotating vapor duct. Be sure that all of the ground glass joint is wetted by the solvent.
 - c. Lower the beaker of solvent until part of the opening of the vapor duct is above the surface of the solvent.

- d. Close the vacuum stopcock.
- e. Gradually raise and lower the beaker to allow the duct to suck up enough solvent to coat the end of the tube.
- f. Partially open the stopcock valve; submerge the rotating vapor duct in the beaker of solvent and adjust the stopcock valve until the vapor duct fills with solvent (but does not overflow). Allow the solvent to remain in the rotating tube for at least 5 seconds. Close the stopcock and allow some of the solvent to overflow into the rotary evaporator receptacle.
- g. Empty the receiving flask and cover the exposed portion of the vapor duct with aluminum foil.

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Study Director Quality Assurance Director

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LOI 6 SUPERCRITICAL FLUID CHROMATOGRAPHY/MASS SPECTROMETRY (SFC/MS)

This LOI describes the SFC/MS instrumentation and methodology for analysis of extracts of OB/OD samples. Preparation of extracts are described in separate LOIs. Three complementary SFC/MS analyses are performed on each extract. Selected-ion monitoring with negative ion chemical ionization (NICI/SIM) is used for the nitroaromatic target analytes. Selected-ion monitoring with positive ion chemical ionization (PICI/SIM) is used for the remaining target analytes. Full scan electron impact ionization (EI/MS) is used to identify nontarget analytes that may be of interest.

A. EI/MS

1. The instrument used for this method is a Lee Scientific Model 602/- Finnigan-MAT Incos 50.
2. The instrument is mass calibrated daily using perfluorotributylamine (FC-43) in the electron impact positive ion mode. Mass calibration is performed using software and recommended procedures provided by the manufacturer.
3. The instrument is tuned in the positive ion mode using FC-43 to optimize sensitivity while maintaining mass resolution.
4. Chromatographic separation involves a Lee Scientific Model 600 SFC. A direct probe interface equipped with a heated frit restrictor delivers the eluent from the SFC to the MS. One microliter of sample is injected using a solvent venting technique. A 5% phenyl methylpolysiloxane stationary phase coated in a 0.050-mm i.d. fused silica capillary column is used for separation. The initial fluid density and temperature is a function of the sample solutes. Density programming is used for analysis, and all conditions (SFC and MS) are identical for all analyses.

5. A standard containing the following analytes is analyzed at the beginning of each day:

- (a) 2,4,6-Trinitrotoluene
- (b) 2,4-Dinitrotoluene
- (c) 2,6-Dinitrotoluene
- (d) 4-Nitrophenol
- (e) 2-Nitronaphthalene
- (f) 1-Nitronaphthalene-d₇ (internal standard)
- (g) 1,3,5-Trinitrobenzene
- (h) 1-Nitropyrene
- (i) Dibenzofuran
- (j) N-Nitrosodiphenylamine
- (k) Benzo[*a*]pyrene
- (l) Benz[*a*]anthracene
- (m) Naphthalene
- (n) 9-Phenylanthracene (internal standard)
- (o) Pyrene
- (p) Biphenyl
- (q) Phenanthrene

Analyte concentration in the standard is approximately 1 ng μL^{-1} for each compound.

6. Calibration curves for each compound are generated by the analysis of a set of standards prepared at approximately 100, 10, 1, 0.1, and 0.01 ng μL^{-1} for each component (see Section E).
7. An analytical blank is periodically analyzed. The blank contains the internal standards f and n.
8. Data acquisition consists of repetitive scanning from *m/e* 100 to *m/e* 500 with a cycle time of approximately 1 sec. Data acquisition begins following elution of the solvent and continues throughout the chromatographic separation process.

9. Mass spectral interpretation is conducted for the most intense chromatographic peaks.
10. No quantitative analysis is performed on the EI/MS data.

B. PICI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 602/- Finnigan-MAT Incos 50.
2. The instrument is mass calibrated and tuned as described in A.2-A.3, except that tuning is conducted in a positive chemical ionization mode.
3. A standard of analytes i to q in A.5 is analyzed daily.
4. A response curve is generated by analysis of the standards at 0.1, 1.0, 10, 100, and 1,000 $\text{pg } \mu\text{L}^{-1}$ of each compound. The internal standard is present in each standard at 240 $\text{pg } \mu\text{L}^{-1}$. The response curve consists of the response of a particular analyte relative to that of the internal standard, plotted as a function of analyte concentration. The purpose of the response curve is to provide quality assurance of response linearity. Quantitative calibration is performed using a standard solution analyzed at the beginning of each day samples are analyzed.
5. Response factors are established for each analyte (i) from analysis of a daily standard solution. The response factor (RF_i) is defined as follows:

Equation (1) The Response Factor

$$\text{RF}_i = \frac{[A_i] [C_{IS}]}{[A_{IS}] [C_i]}$$

where A_i and A_{IS} are the selected ion chromatographic peak areas of the analyte and internal standard, respectively. C_i and C_{IS} are the concentrations of the analyte and internal standard, respectively.

6. OB/OD samples and field controls are analyzed using the same instrumental conditions as the standard. Identification of target analytes are by retention time and response at the specific mass monitored for each analyte. The concentration of each analyte (C_i) as mass per unit volume of extract (eg. ng/mL) is defined as:

Equation (2) Analyte Concentration

$$C_i = \frac{[A_i] [C_{IS}]}{[A_{IS}] [RF_i]}$$

where the variables are functionally equivalent to those in B.5 but whose values are obtained from the analytical data of the OB/OD sample.

C. NCI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 602/-Finnigan-MAT Incos 50.
2. The instrument is mass calibrated as in A.2.
3. The instrument is tuned in the negative ion mode using FC-43 to optimize sensitivity while maintaining unit mass resolution. Tuning does not affect mass calibration.
4. A standard of analytes a to h in A.5 is analyzed daily.
5. The remainder of the analytical procedure is equivalent to that described in B.4-B.6.

D. Data Reporting and Archiving

1. The results from each EI/MS analysis consist of the following:

- (a) A total ion chromatogram annotated to indicate the peaks identified.
- (b) A background corrected mass spectrum for each identified compound.
- (c) A table consisting of a spectrum number and identification.

Where possible, an order of magnitude estimation of concentration is included.

2. The results from each NICI/SIM/MS and PICI/SIM/MS consist of the following:

- (a) A single page reconstructed (total ion) chromatogram.
- (b) A single page mass chromatogram containing the quantification area for each analyte and internal standard.
- (c) A table containing the response factors for each analyte and internal standard.

3. For each sample, a summary table is provided containing all target analytes found with final concentrations expressed as ng/sample.

4. Raw SFC/MS data are archived in disk image format on data cartridge tapes (DC 30 XL/p) at 10,000 SApi.

E. Determination of Calibration Curves and Detection Limits for SFC/MS

- 1. A stock solution of the target organic analytes in methylene chloride at a concentration of 100 ng μL^{-1} is prepared.
- 2. A logarithmic dilution of the stock solution (1 to 10 dilution) is made until the signal observed in the reconstructed total-ion chromatogram for the analytes in the mass spectrometer is less than three times the background signal.

3. The signal observed from the INCOS 50 data system is given in relative ion counts (RIC).
4. The point at which the RIC for the analytes is three times that of the background corresponds to the detection limits.
5. A graph of concentration versus observed peak area is made to construct a calibration curve for each analyte. This serves as a standard curve and is sometimes used to determine concentrations in unknown samples. This is accomplished by matching the observed signal to the corresponding concentration for that analyte. Where possible all quantitations are made from response curves and comparison to the internal standards.
6. Limits of quantification refer to the lowest concentration detectable above the background (analyte RIC at three times the background RIC).
7. All of the compounds on the target organic analyte list are tested in a like manner.
8. Steps 1 to 7 are used for both electron impact and chemical ionization.

Target Organic Analytes

- | | |
|--|--------------------------------------|
| 1. 2,4-Dinitrotoluene ^{a,b} | 2. 2,6-Dinitrotoluene ^{a,b} |
| 3. 2,4,6-Trinitrotoluene ^{a,b} | 4. 2-Nitronaphthalene ^{a,b} |
| 5. N-Nitrosodiphenylamine ^{a,b} | 6. 2-Nitrodiphenylamine ^b |
| 7. 4-Nitrodiphenylamine ^b | 8. Nitroglycerin ^b |
| 9. 1,3,5-Trinitrobenzene ^a | 10. 1-Nitropyrene ^a |
| 11. Naphthalene ^{a,b} | 12. Benz[a]anthracene ^{a,b} |
| 13. Benzo[a]pyrene ^{a,b} | 14. Pyrene ^b |
| 15. Phenol ^{a,b} | 16. Dibenzofuran ^{a,b} |
| 17. Diphenylamine ^{a,b} | 18. Diethyl Phthalate ^b |

^aAnalytes for open detonation

^bAnalytes for open burning

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Quality Assurance Director

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ALPINE WEST

LOI 7 QUALITY CONTROL PLAN: PROCEDURES FOR ACCURACY, PRECISION, AND COMPLETENESS.

A. Weighing

1. All balances are calibrated monthly by a Sartorius service engineer.
2. The analytical balance (Model 2434) is accurate to ± 0.00001 g, and the top-loading balance (Model E5500S) is accurate to ± 0.01 g.
3. All samples are weighed 3 times.
4. Blank samples are weighed periodically to monitor the fluctuation in sample weights.

B. Extraction

1. Filters

- a. Extraction efficiency of analytes from filters is calculated by the analysis of solutions from the extraction of 6 filters spiked with known concentrations of target analytes. Three filters are spiked in triplicate with a high concentration and three with a low concentration.
- b. Completeness of extraction is measured by (1) re-extraction of the high spiked filters and (2) the second extraction of a real test filter picked randomly.
- c. During the extraction of real samples, approximately 10% additional filter samples will be introduced as blanks.

2. Soils and Particulates

- a. Extraction efficiency of analytes from soil is calculated by the analysis of solutions from

the extraction of 6 soils spiked with known concentrations of target analytes. Three soils are spiked in triplicate with a high concentration and three with a low concentration.

- b. Completeness of extraction is measured by (1) re-extraction of the high spiked soils and (2) a second extraction of a real test soil picked randomly.
- c. Five audit samples (spiked by the EPA) are extracted to determine the accuracy of the method.
- d. During the extraction of real samples, approximately 10% additional soil samples are introduced as blanks.
- e. One test soil is split and analyzed by standard addition of the target analytes.

C. Sample Storage

- 1. The temperature of the freezer used for storage is monitored daily.
- 2. The effect of storage on the samples is monitored as follows:
 - a. Six spiked filters, 6 spiked soils, and 6 acetonitrile standard solutions are placed in the freezer.
 - b. After one day storage, 2 filters, 2 soils, and 2 acetonitrile solutions are removed and analyzed.
 - c. After approximately 15 days storage the second pairs of samples are analyzed.
 - d. After all test samples have been analyzed, the last pairs of storage samples are extracted and analyzed.

D. SFC/MS and GC/MS

1. Instrument calibrations with FC-43 are run daily and logged.
2. Analyte standard solutions are run daily, and peak areas relative to the internal standards are calculated. A deviation of over $\pm 10\%$ will signify a dirty system and require cleaning.
3. Matrix blanks are run at the approximate frequency of 1 in every 10 field samples ($\sim 10\%$).
4. All samples are run in duplicate.

E. Sampling Handling

1. All samples are given an internal identification number.
2. All sample collection (ELI) sheets are checked against samples for completeness and correctness.
3. All samples are logged as to where they are stored and where they came from.
4. All samples have an AWL analysis sheet assigned (see attached).

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Study Director Quality Assurance Director

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ALPINE WEST

LOI 8 PREPARATION AND HANDLING OF 32-L TANK EXTRACTS

A. Sample Preparation

Each 450-900 mL methylene chloride extract obtained from OGC is concentrated to a volume just under 1 mL using a rotary evaporator (30°C), transferred to a 1-dram amber vial (Teflon™-lined cap), and brought to a volume of 1 mL by adding nanograde methylene chloride. The sample is then split into two equal halves, one half spiked with 100 mL of an internal standard (300 pg of 1-nitronaphthalene-d7 and 240 pg of 9-phenylanthracene).

B. Storage of Methylene Chloride Extracts

The 1-dram amber vials containing the final tank extracts are stored at -20°C in a freezer.

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ALPINE WEST

LOI 9 ANALYSIS OF THE EXTRACT FROM THE SPIKED 32-L TANKS

Two 32-L tanks used by OGC in the preliminary test at SNL will be spiked by the US EPA with exotics.

1. The extract from the 32-L tanks, obtained from OGC, will be split into three parts; one part to be sent to BCD, one part to US EPA and one retained for analysis.
2. The extract part is then concentrated to a volume of 4 mL by rotary evaporation and placed in a Teflon™-sealed glass bottle.
3. The extract will be analyzed by both SFC/MS and GC/MS.

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ALPINE WEST

LOI 10 ANALYSIS OF THE 32-L TANK EXTRACTS

The OGC will sample the explosive plume with 32-L tanks to collect the exotics. OGC will then reflux the tanks, collect the extract, and send this extract to AWL.

1. The 32-L tank extract will be concentrated to a volume of 4 mL by rotary evaporation.
2. This concentrated sample will then be split into two equal parts; one sent to BCD and one retained for analysis.
3. The 2 mL sample will be kept in a 4 mL Teflon™TM-sealed glass bottle and analyzed by SFC/MS.

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ALPINE WEST

LOI 11 PREPARATION AND HANDLING OF XAD-2™ RESIN TRAPS, FILTERS, AND VOST

A. Preparation and Storage of Resins

1. Polystyrene divinylbenzene polymer beads (XAD-2™ purified, 20/50 mesh) are purchased from Alltech Associates, Inc., in 100-g bottles.
2. Resin (200-g portions) is extracted using nanograde (Baker) acetonitrile (500 mL) in a Soxhlet extractor (extra large 1000-mL round bottom flask, and 60-mm x 180-mm cellulose thimble) for 12 h using a heating mantle and variable power transducer to provide a solvent temperature of 80°C.
3. After extraction, the resin is placed in a clean glass bottle (1000-mL) and dried at room temperature under an argon gas purge for approximately 15 min.
4. The dry resin is stored in clean 32-oz. amber bottles (Teflon™ TM-lined caps) at room temperature.

B. Preparation of Glass Resin Cartridge Holders

1. Glass resin cartridge holders are constructed as illustrated in Figure 1.
2. All components, except the quartz wool, are washed with hot tap water and detergent, rinsed with distilled water, placed in a concentrated nitric acid bath for 10 min, rinsed again with distilled water, placed in a 10% KOH/isopropanol base bath for 10 min, rinsed again with distilled water, and finally fired in a muffle furnace at 650°C for 8 h. The cleaned cartridge holder components are stored in clean 32 oz. glass bottles (Teflon™ TM-lined lids).

3. The resin cartridge holders are assembled by fixing the 3 stainless-steel screens (two coarse and one 200-mesh) in place by bolting together as shown in Figure 2. A layer (1/8 in.) of quartz wool is placed on top of the screen pad. Clean XAD-2™ resin (either 65-g or 20-g portions weighed accurately to 0.01 g using a top-loading balance) is firmly packed on top of the quartz wool by careful tamping, followed by another layer (1/8 in.) of quartz wool,

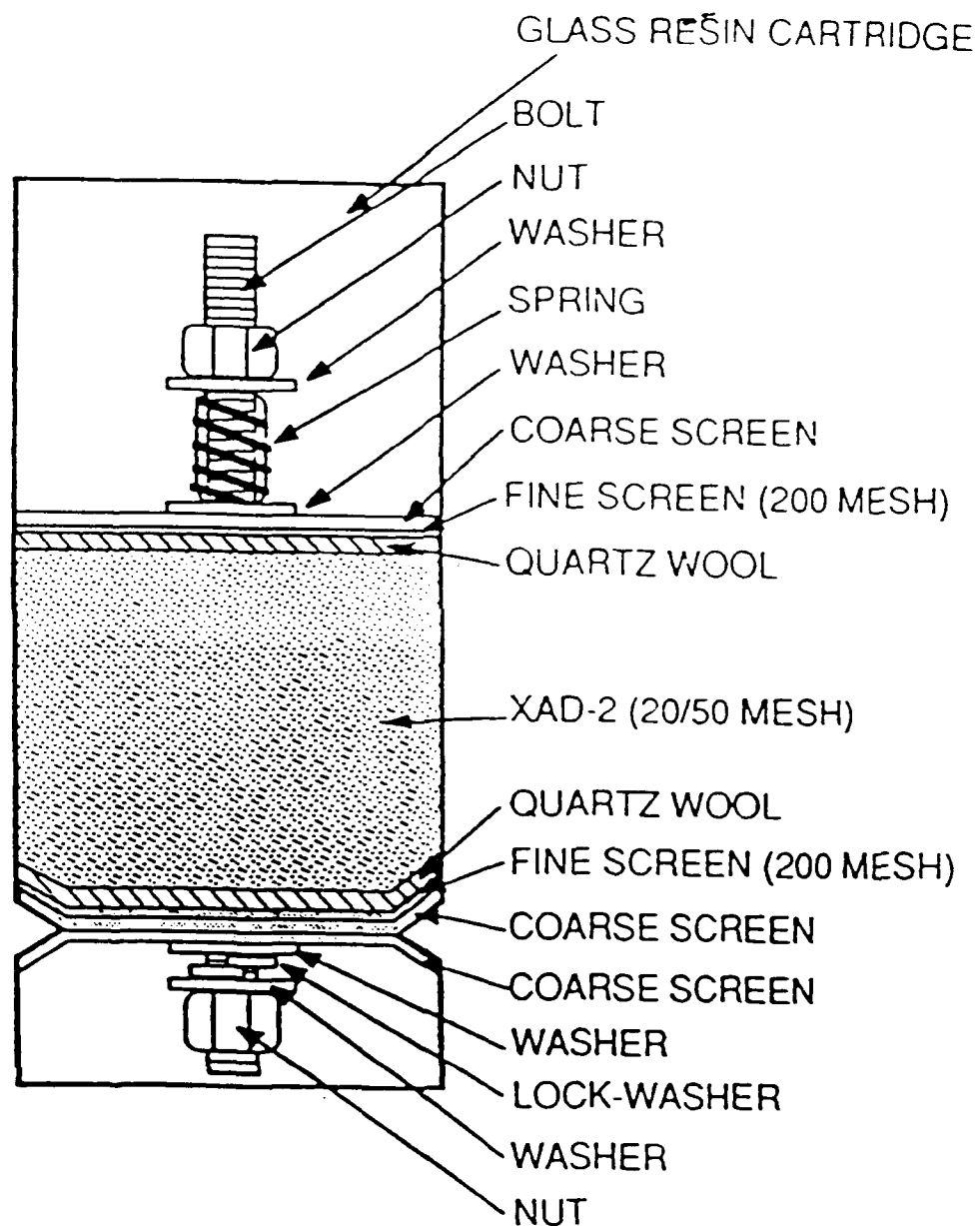


Figure 1. XAD-2™ Resin Trap.

a 200 mesh stainless-steel screen, and a coarse stainless-steel screen. A washer, spring, and second washer ensure that the packed bed remains physically stable.

4. The assembled XAD-2™ resin traps are stored at room temperature in clean 32-oz. glass bottles (Teflon™-lined lids) at all times, except during sampling.

C. Preparation of VOST Trains

1. The VOST train consists of three sections: a top filter section followed by two resin sections
2. The VOST train is assembled on a clean surface and rinsed with nanograde (Baker) methylene chloride. Cotton gloves are worn at all times to decrease the chance of contamination.
3. The 10.2-cm quartz fiber filter is placed with tweezers between two Teflon™ rings to create a tight seal. Wire screens are placed above and below this to protect against filter breakage during the blast. This all fits under the cap on top of the VOST train.
4. The 65-g XAD-2™ resin cartridge is removed from its storage jar and placed in the top trap with a rubber gasket on the bottom. The VOST train's threads are wrapped with Teflon™ tape.
5. Placement of the 20-g XAD-2™ resin cartridge follows the same procedure, but it is placed in the bottom trap.
6. Each VOST train is labeled as to its particular use during the test (VOST number, time, location) and aluminum foil is placed over the bottom entrance to keep out contaminants. A steel cap seals the top.

D. Removal of Samples from VOST Trains

1. The VOST train is disassembled on a clean surface with cotton gloves.
2. The 10-cm quartz fiber filter is removed with Teflon™-coated tweezers. It is folded over on itself, and then placed in an aluminum foil bag, which is then placed in a zip-loc bag and labeled appropriately.
3. The 65-g XAD-2™ resin cartridge is removed from the top trap in the VOST train and placed immediately into its storage bottle. The bottle is lined with aluminum foil to cushion the cartridge during travel. Strapping tape is placed over the bottle lid to insure that the top will not come loose. A label is placed immediately on the bottle which identifies the sample.
4. The 20-g XAD-2™ resin cartridge is removed from the bottom trap in the VOST train and treated according to the same procedures as the 65-g XAD-2™ cartridge.

E. Preparation of Samples for Transport

1. Boxes (cardboard) with dividers that hold a maximum of 12 sample bottles are used. Usually the two middle holes are vacant so a travel "blank" and the filter samples can be sent.
2. The ELI numbered collection sheet of all samples present in the box are packed in the box, on top of the samples.
3. The cardboard box is placed in a vinyl-coated canvas carry bag which zips shut.

F. Transportation

The bag/box serves as carry on luggage by person traveling to respective labs.

G. Extraction of Resin Traps After Sampling

1. The assembled glass resin cartridge is placed inside a Soxhlet extractor (Fig. 1; extra large, 1000-mL round bottom flask) and is extracted with 500-mL of nanograde (Baker) acetonitrile for 6 h at a solvent temperature of 80°C.
2. The acetonitrile extract is concentrated to a volume just under 1 mL using a rotary evaporator (50°C), transferred to a 1-dram amber vial (Teflon™-lined cap), and brought to a volume of 1 mL by adding nanograde acetonitrile. The extract is filtered to remove any resin beads.
3. The extract is divided in half and an internal standard (300 pg of 1-nitronaphthalene-d7 and 240 pg of 9-phenylanthracene) is placed in one of the vials. Both are placed in storage.

BATTELLE-COLUMBUS DIVISION

LOI 1 SAMPLE PREPARATION FOR GC/MS AND STORAGE OF OB/OD SAMPLES

1.0 Scope

This LOI describes log-in, storage and preparation of OB/OD samples prior to analysis using Gas Chromatography/Mass Spectrometry (GC/MS). Sample analysis is described in a separate LOI (BCD-OB/OD-2). Samples types and anticipated number of each type are indicated below:

- (a) Acetonitrile extracts of resins and filters (37)
- (b) Spiked and blank soil samples (5)
- (c) Spiked and blank XAD™ resin samples (3)
- (d) Spiked and blank Porapak-R™ resin samples (3)
- (e) Acetone reference solution (1)
- (f) Methylene chloride reflux extracts of 32-liter tanks (4)

2.0 Sample log-in and storage

All samples received will be logged and inspected by the principal investigator or his designated representative. Information logged will include:

- (a) Date and time of receipt
- (b) Count and identity of samples in the shipping container
- (c) Visual condition of each sample
- (d) Estimated temperature of shipping container interior

Samples will be stored in an explosion proof refrigerator at approximately 4 °C or in a freezer at approximately -60 °C. Samples stored at -60 °C will be the acetonitrile extracts of resins and filters, and the acetone spiking solution. All other samples will be stored at 4 °C.

3.0 Glassware and laboratory environment

Glassware will be solvent rinsed and placed in an explosion prove furnace at approximately 450 °C for a period of 8 to 16 hours. Extraction and other sample preparation will be conducted to the extent practical in a laboratory equipped with yellow fluorescent lights in order to minimize possible degradation of light sensitive compounds.

4.0 Preparation of acetonitrile extracts

4.1 Prior to use, each acetonitrile extract will be warmed to room temperature and weighed to the nearest 0.01 g.

4.2 The contents of the acetonitrile vial will be transferred, then rinsed into an appropriately sized round bottom flask using reagent acetonitrile. The sample vial and cap will be air dried and weighed to the nearest 0.01 g. The difference weight and volume (based on the literature density of acetonitrile) will be recorded.

4.3 The acetonitrile sample will be evaporated to near dryness under vacuum using a rotary evaporator. Reagent toluene will be added and the mixture further evaporated to near dryness under vacuum to remove residual acetonitrile and water which may have been present in the sample.

4.4 The contents of the rotary evaporator flask will be rinsed into a graduated tapered glass vessel with reagent toluene. The volume will be adjusted to 1.0 ml by evaporation with a stream of dry, high purity nitrogen.

4.5 The sample will be spiked with internal standards d12-perylene, d7-nitronaphthalene, and 9-phenylanthracene. The quantity spiked will be determined from preliminary trials.

4.6 The sample will be removed from the graduated tapered vessel with a disposable pipette and placed in a Teflon™ lined screw cap vial. The vial will be suitably labeled and stored at -60 °C until analysis.

5.0 Preparation of Soil Samples

5.1 Each sample will be warmed to room temperature. The entire sample will be transferred to a suitably sized glass jar, sealed with a Teflon™ lined screw cap and homogenized by tumbling or rotation.

5.2 A 25-g aliquot will be removed and weighed to the 0.01 g. The remainder of the sample will be replaced in its original container and stored. The aliquot will be soxhlet extracted with approximately 300 ml of methylene chloride or sonicated with a comparable volume of acetonitrile. The extraction technique will be selected based on preliminary trials.

5.3 If samples are sonicated, the procedure described in 4.3-4.6 will be followed. Otherwise the sample will be reduced in volume using a combination of Kuderna-Danish and dry nitrogen evaporation.

5.4 The methylene chloride extract will be spiked with internal standards at a level to be determined. Extracts will be labeled and stored at -60 °C until required for analysis.

6.0 Spiked and blank XAD-2™ resin samples will be prepared as described in 5.1-5.4.

7.0 Spiked and blank Porapak-R™ resin samples will be prepared as described in 5.1-5.4.

8.0 Acetone Reference Solution

8.1 Based on the approximate concentration provided, the acetone reference solution will be concentration adjusted by nitrogen evaporation or dilution in order to match the approximate working range of the GC/MS systems.

8.2 The reference solution will be spiked with internal standards and maintained at -60C until required for analysis.

9.0 Methylene chloride reflux extracts from the 32-liter tanks will be prepared as described in 5.3- 5.4.

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BATTELLE-COLUMBUS DIVISION

LOI 2 GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) ANALYSIS OF OB/OD SAMPLES

1.0 Scope

This LOI describes the GC/MS instrumentation and methodology for analysis of extracts of OB/OD samples. Preparation of extracts is described in a separate LOI (BCD-OB/OD-1). Three complimentary GC/MS analyses will be performed on each extract. Selected ion monitoring with negative chemical ionization (NCI-SIM) will be used for the nitroaromatic target analytes. Selected ion monitoring with positive chemical ionization (PCI-SIM) will be used for the remaining target analytes. Preliminary trials conducted prior to analysis of any OB/OD samples may warrant the substitution of selected ion monitoring electron impact ionization (EI-SIM) for PCI-SIM. Full scan electron impact ionization (EI-MS) will be used to identify non-target analytes that may be of interest to the OB/OD study.

2.0 NCI-SIM

2.1 The instrument used for this effort will be a FinniganTM TSQ-45 GC/MS/MS system (tandem mass spectrometer) operated in a conventional GC/MS mode. A backup instrument for this method will be a Finnigan TM 4500 GC/MS system.

2.2 The instrument will be mass calibrated daily using perfluorotributylamine (FC-43) in an electron impact positive ion mode. Mass calibration will be performed using software and recommended procedures provided by the manufacturer.

2.3 The instrument will be tuned in the negative ion mode using FC-43 to optimize sensitivity while maintaining unit mass resolution. Tuning will not affect mass calibration.

2.4 Chromatographic separation will involve a 1- to 2-microliter split less injection followed by temperature programming of a bonded phase 5% phenol methyl silicone fused silica capillary

column. The initial column temperature will be a function of solvent (methylene chloride, acetone, toluene or acetonitrile) boiling point. All other conditions (GO and MS) will be identical for all analyses.

2.5 A standard of the following analytes will be analyzed at the beginning of each day that analyses are performed:

- (a) 2,4,6-Trinitrotoluene
- (b) 2,4-Dinitrotoluene
- (c) 2,6-Dinitrotoluene
- (d) 4-Nitrophenol
- (e) 1,6-Dinitropyrene
- (f) 2-Nitronaphthalene
- (g) d7-Nitronaphthalene (internal standard)

Analyte concentrations in the standard will be approximately 10 times the instrument quantification limit for each compound. Quantification limits will be estimated from preliminary trials conducted prior to analysis of OB/OD samples.

2.6 A response curve will be generated by analysis of a set of standards prepared at 0.3, 1, 3, 10, and 30 times the estimated quantification limit. The internal standard will be present in each standard at 10 times the estimated quantification limit. The response curve will consist of the response of a particular analyte relative to that of the internal standard, plotted as a function of analyte concentration. The purpose of the response curve is to provide quality assurance of response linearity. Quantitative calibration will be performed using a single point standard analyzed at the beginning of each day samples are analyzed.

2.7 Response factors will be established for each analyte (i) from analysis of a daily single point standard. The response factor (RF_i) is defined as follows:

Equation (3) Response Factor

$$RF_i = \frac{(A_i) (C_{IS})}{(A_{IS}) (C_i)}$$

where A_i and A_{IS} are the selected ion chromatographic peak areas of the analyte and internal standard respectively.

C_i and C_{IS} are the concentrations of the analyte and internal standard, respectively.

2.8 A laboratory blank will be analyzed once each day containing only the internal standard. Evidence of target analytes at or above the estimated quantification limit may necessitate replacement of the injector septum, injector liner, syringe, or removal of the first one or two meters of the fused silica capillary column.

2.9 OB/OD samples and field controls will be analyzed using the same instrumental conditions as the standard. Identification of target analytes will be by retention time and response at the specific mass monitored for each analyte. The concentration of each analyte (C_i) as mass per unit volume of extract (e.g. ng/mL) will be defined as:

Equation (4) Analyte Concentration

$$C_i = \frac{(A_i) (C_{IS})}{(A_{IS}) (RF_i)}$$

where the variables are functionally equivalent to those in 2.7 but whose values are obtained from the analytical data of the OB/OD sample.

2.10 The extract concentration data will be extrapolated to air concentration data (ng/m^3) in the case of semi-VOST resins, filters and 32-liter tank samples. Extrapolation will take into account the total air volume sampled, in cubic meters (V) by the matrix, the fraction of matrix extracted (FE), and the fraction of extract spiked with internal standards (FS). The airborne (vapor or particulate bound) concentration (ng/m^3) would be represented as:

Equation (5) Airborne Vapor or Particulate Concentration

$$C_i(\text{air}) = \frac{(C_i) (v)}{(FE) (FS) (V)}$$

where v is the volume (ml) of extract spiked with internal standards. In the case of spiked soils, resins and reference solution, the extract concentration data will be extrapolated to ng/g of matrix

or ng/g of solution as appropriate. If the reference solution is concentration adjusted prior to the addition of internal standards, the fractional change in volume will provide an additional factor in the extrapolation. For series configured devices sampling the same air volume (filter, resin, backup resin) numerical summation of the individual air concentrations will provide a net air concentration for a particular analyte.

2.11 A control standard or repeat analysis of a given extract (analytical duplicate) will be conducted once each day. The analytical duplicate will be selected from samples analyzed on the same day.

3.0 PCI-SIM

3.1 The instrument used for this method will be a Finnigan 4500 GC/MS system. A backup instrument for this method will be a Finnigan TSQ-45 or a second Finnigan 4500 GC/MS.

3.2 The instrument will be mass calibrated and tuned as described in 2.2.2.4 except that tuning will be conducted in a positive chemical ionization mode.

3.3 A standard of the following target analytes will be analyzed daily:

- (a) Phenol
- (b) Benz[a]anthracene
- (c) Benzo[a]pyrene
- (d) Dibenzo[a,h]anthracene
- (e) Benz[c]acridine
- (f) Dibenzofuran
- (g) 2-Naphthylamine
- (h) N-nitrosodiphenylamine
- (i) d12-Perylene

3.4 The remainder of the analytical procedure is equivalent to that described in 2.6-2.11.

4.0 EI-MS

4.1 The instrument used for this method will be a Finnigan 4500 GC/MS system. Backup instruments will be either a second Finnigan 4500 GC/MS or a Finnigan TSQ-45.

4.2 The instrument will be mass calibrated and tuned as described in 2.2-2.4 except that tuning will be in an electron impact ionization mode.

4.3 An analytical blank will be analyzed at the beginning of each day that samples are analyzed. The blank will contain the internal standard 9-phenylanthracene.

4.4 There are no target analytes associated with this method. The instrument sensitivity will be based on the response of the internal standard in the analytical blank.

4.5 Data acquisition will consist of repetitive scan from m/z 40 to m/z 450 with a cycle time of approximately 0.75 seconds. Data acquisition will begin following elution of the solvent and continue throughout the chromatographic separation process.

4.6 Chromatographic peaks will be selected subjectively for mass spectral interpretation by the principal investigator or another BCD professional mass spectrometrist. Selection will be based primarily on the intensity and quality of the background corrected mass spectra at the chromatographic peak maxima.

4.7 Mass spectral interpretation will be conducted, where possible, for the most intense chromatographic peaks up to a maximum of 20.

4.8 Mass spectral interpretation will be driven primarily by visual evaluation of the data by a BCD professional mass spectrometrist. The mass spectrometrist may supplement his interpretation with various tools including an on-line library search of the NBS (NIST) mass spectral data base and published reference volumes of mass spectra.

4.9 Identifications may be confirmed for a limited number of compounds of greatest interest to

the OB/OD technical steering committee. Confirmation will involve preparation of solutions containing authentic reference compounds with 9-phenylanthracene internal standard. Confirmation will be indicated by equivalence of mass spectra and retention time relative to 9-phenylanthracene.

4.10 An order of magnitude estimate of concentration for each identified compound (i) in the extract will be made as follows:

Equation (6) An Order of Magnitude Estimate of Concentration

$$C_i = \frac{(TIC_i) (C_{IS})}{(TIC_{IS})}$$

where TIC_i is the total ion current represented by the background subtracted mass spectrum of the identified compound. TIC_{IS} is the comparably obtained value for the internal standard spectrum, and C_{IS} is the concentration of the internal standard.

5.0 Reporting and Data Archive

5.1 The results of each NCI-SIM and PCI-SIM will consist of:

- (a) a single page reconstructed (total ion) chromatogram annotated to identify the chromatographic peaks of the target analytes, and

(b) a single page quantitation report containing the quantification masses, areas, retention times and response factors for each analyte and internal standard, as well as concentration in ng/MS of extract.

5.2 The results from each EI-MS analysis will consist of:

- (a) a total ion chromatogram annotated to indicate the peaks identified,
- (b) a background correct mass spectrum for each identified compound, and
- (c) a single page table consisting of a spectrum number, identification and a subjective measure of confidence. Where an identification is not possible on a major component, functionality, molecular weight and substructure features will be provided to the extent possible. An order of magnitude estimation of extract concentration will be included.

5.3 For each sample, a summary table will be provided summarizing all target analytes found with final concentration expressed as ng/m₃ or ng/g as appropriate.

5.4 Raw GC/MS data will be archived in disk image format on 9-track magnetic tape at 800 or 1600 bpi.

Approved: _____ Received: _____
Study Director Quality Assurance Director

Date Date

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SANDIA NATIONAL LABORATORY

LOI 1 EXPLOSIVE PREPARATION FOR OB/OD BANGBOX TRIALS

1. During the Bang Box trials at Sandia Labs (SNL), a 1/2 pound block of TNT will be the explosive. The TNT is from lot # 10P85K027001. This material came from Tooele Army Depot (TEAD). It was drawn from U.S. Army stock and meets Mils Specs.

2. SNL will provide the blasting caps. The caps required by SNL safety personnel are exploding bridge wire (EBW). The EBW is of a known composition. The chemical formula for the EBW will be given to the PM for use in analytical work. The EBW is fired from a capacitor that is charged to 3000 volts.

3. The TNT block will be armed, suspended, and fired by explosive handlers IAW the following procedures.

a. Explosive Preparation

1. Select TNT block.

a. Block to be in enclosed casing.

b. Note and record the lot number to insure the same lot is used for all trials.

2. Remove block to preparation site.

3. Carefully remove the cap end plate with a knife.

a. Cut around the four sides.

b. Cut next to the metal end to avoid physical damage to the explosive.

4. Remove the explosive from the can and place on a clean sheet of paper.
5. Tie secure-and-tight wraps around the block using monofilament line. The wraps must go both ways around the long faces of the block.
6. Use monofilament to tie a knot that will secure the wraps around the block on the top face. Use a long strand of line.
7. Tie two pieces of line (2m each) to the wraps on opposite sides of the top face.
8. Insert the EBW into the cap well and secure by tying a knot into the wraps as in

b. Explosive Mounting

1. Insure that all work in the air building is complete.
2. Remove TNT from preparation area and carefully move to air building.
3. Position the block near the center of the stand with cap well facing down.
4. Thread one 2-m line through the hole near the top of an adjacent rod.
5. Thread the opposite strand through the hole in the opposite rod.
6. Adjust strands so that the charge is centered and is one meter above the stand base. Tie the lines off to the bolts on the base to maintain the proper alignment.
7. Tie the long line on the base of the charge (step a-8) to the closest, opposite support rods.
8. Tie the long line on top of the charge (step a-6) to the support rods.

c. Explosive Priming: Follow SNL's established SOP to connect the blasting cap to the firing wire.

d. Explosive Initiation.

1. Follow SNL's established SOPs to prepare for initiation.

2. When using a high speed camera, initiate the cap at the instant the camera reaches the preset speed.

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LOI 2 TRINITROTOLUENE (TNT) DETONATION

1.0 Purpose

This instruction is intended to cover all aspects of the TNT detonation trials conducted at Sandia National Laboratories (SNL) and the gas and aerosol instrument calibration procedures that fall under the responsibility of the SNL test group. The instructions cover all aspects of instrument setup and calibration, sample collection, data processing and analysis, as well as a detailed description and check list of the steps to be followed during each detonation in the inflatable structure.

2.0 Test Description

Three tests will be conducted at the SNL air building in which detonation products of TNT will be sampled and analyzed in detail. In each test a 0.5-pound quantity of TNT will be detonated inside the structure. Following detonation, gas and aerosol sampling techniques will be used to sample and analyze the detonation products for an extended period of time following the detonation. Complete details concerning the instruments deployed during these tests are given in the Test Plan.

3.0 Real-time instrument calibration

3.1 Gas instruments

All gas instruments to be deployed in this test series will be calibrated both before and after the test series. The level of significance associated with the data from these various instruments and how that data contributes to the overall objectives of the test will determine the degree of effort given to calibration for each instrument. Specifics for each instrument follow:

3.1.1 Oxides of nitrogen (NO_x) instrument

A multi-point calibration will be carried out on the NO and NO_x channels of the instrument using

standard procedures outlined in Appendix A. This calibration will be carried out prior to and following the entire test series. A NBS (NIST)-traceable certified standard NO test gas will be diluted with zero air using a gas dilution system to produce 5 separate gas concentration levels in the working range of the instrument. The voltage response of the instrument over its working range will be determined by simple linear regression analysis of the calibration data as outlined in Appendix B. The resultant slope and intercept of the regression line will be used to convert instrument voltage output to engineering units during data processing.

Daily checks of instrument performance will be carried out using a zero gas and a NO span gas with a concentration in the working range of the instrument. In the event that the recorded instrument output during a span gas check deviates from the expected output by more than 20 percent, a multi-point calibration will be repeated prior to continuation of the test series.

3.1.2 Carbon monoxide instrument

Calibration procedures identical to those carried out for the NO_x instrument will be followed for the CO instrument using a certified CO test gas.

3.1.3 Carbon dioxide instrument

Calibration procedures identical to those carried out for the NO_x instrument will be followed for the CO₂ instrument using a certified CO₂ test gas.

3.1.4 Sulfur dioxide instrument

Calibration procedures identical to those carried out for the NO_x instrument will be followed for the SO₂ instrument using a certified SO₂ test gas.

3.1.5 Ozone instrument

A multipoint calibration of the O₃ instrument will be conducted prior to the test series using a certified ozone calibration source maintained and operated by the City of Albuquerque Air

Pollution Monitoring Division. Daily zero checks will be carried out on the ozone instrument prior to and following each test. Due to the unavailability of a calibrated ozone source, daily span checks on this instrument will not be carried out.

3.1.6 Hydrocarbon instrument (FID)

Zero checks will be carried out on this instrument by placing a charcoal scrubber on the instrument inlet. A one-point span check will be performed using cylinder contained 10 ppm of methane in air.

3.1.7 Hydrocarbon instrument (PID)

Zero checks on this instrument will be carried out in accordance with the instrument instruction manual by turning off the UV lamp in the instrument. Span checks will be performed using a cylinder containing 100 ppm isobutylene.

3.2 Aerosol instruments

3.2.1 Real-time aerosol monitor

Zero checks will be carried out in accordance with manufacturers instructions by inserting an absolute aerosol filter on the inlet of the instrument. Span checks will be carried out by inserting an optical scattering device in the optical path of the instrument.

3.2.2 Particle probe (ASASP-100-X)

The performance of this probe will be checked prior to the beginning of the test series by passing polystyrene latex aerosol particles of known size (0.7-and 2.0-micrometer diameter) through the optical path of the instrument. This test will provide a measure of the sizing accuracy of the instrument. Instrument response will be judged acceptable if the particle number count peak channel is within two channels of the expected channel as predicted by the original factory calibration. Laser alignment of the probe will be carried out prior to this calibration using the procedures outlined in the instrument instruction manual. Due to the difficulty of performing a number count calibration

in the field, the most recent (March, 1988) factory calibration will be used for number concentration measurements during this test series.

3.2.3 Particle probe (FSSP-100-X)

The performance of this probe will be checked prior to the beginning of the test series by passing PSL aerosol (2.0, 4.0 micrometer diameter) through the optical path of the instrument. Instrument particle sizing response will be considered acceptable if the maximum particle number count channel falls within two channels of the expected channel as determined by the most recent factory calibration. Due to the difficulty of performing a number count calibration in the field, the most recent (March, 1988) factory calibration will be used for number concentration measurements during this test series.

3.2.4 Particle spectrometer (APS)

Since this instrument will be used for qualitative purposes only during this test series, the most recent factory calibration will be used. No field calibration will be carried out during this test series.

3.2.5 Particle spectrometer (DMPS)

Since this instrument will be used for qualitative purposes only during this test series, the most recent factory calibration will be used. No field calibration will be carried out during this test series.

3.2.6 Nephelometer

Zero checks on this instrument will be carried out by filling the optical chamber with particle free air. Span checks will be carried out using the known scattering properties of Freon-12. Pressure and temperature corrections will be applied to span values published in the literature for Freon-12.

4.0 VOST Sampler Calibrations

VOST sampler calibrations include a complete calibration of all flow measuring devices used in

conjunction with these samplers. Specific details follow:

4.1 Internal VOST sampler flow calibration

A multipoint flow calibration of the VOST samplers to be positioned inside the air building will be carried out prior to the beginning of the test series. A factory supplied calibrated orifice will be used to determine flow rate as a function of pressure drop across an orifice located in the air flow path of the instrument.

4.2 Aircraft system VOST sampler flow calibration

Electronic mass flow meters will be installed in-line with both VOST samplers in the aircraft system. The voltage response as a function of flow rate through these mass flow meters will be determined by a multipoint calibration of each mass flowmeter. Since these mass flow meters provide a linear response of voltage as a function of flow rate, a linear regression of a calibration data will be used to determine the slope and intercept parameters used to convert voltage output to air flow rate through the mass flow meter. For these mass flowmeter calibrations, the reference flow standard used will be either a primary standard flow measuring device (bubble meter) or a secondary flow standard (dry test meter) with calibration data that references its performance to a primary flow standard.

4.3 Internal SEM/XRF filter sampler flow rate calibration

SEM/XRF filter flow rate calibration will be determined by measuring the flow rate through the filter before and after the sampling period with a calibrated dry test meter.

4.4 Aircraft system SEM/XRF filter sampler flow rate calibration

Electronic mass flow meters will be installed in-line with these filter units and will be calibrated in the same manner as outlined in section 4.2.

5.0 Test Sequence Check List

5.1 T-5 hr

All instrument systems turned on 10 VOST cartridges loaded 5 SEM/XRF filter sets loaded Data files created on PMS and Analog data acquisition system Date and time set on the following computers:

APS-IBM

DMPS-IBM

HP200-PMS

HP200-Analog

PDS-400

Metrosonics TM

Program parameters set on the Metrosonics TM data logger

5.2 T-3 hr to T-1 hr

5.2.1 Open calibration file on HP data acquisition system and begin logging gas instrument calibration data.

5.2.2 NO_x instrument

NO full scale: 5 ppm

NO_x full scale: 5 ppm

NO₂ full scale: 0.5 ppm

Time constant: 5 sec

Check to see that clean filter is installed on inlet line.

Check zero and span (electric) on NO_x instrument.

5.2.3 CO instrument

CO full scale: 10 ppm

CO time constant: 10 sec

CO cell pressure less than 800 torr

Check zero and span on CO instrument.

5.2.4 CO₂ instrument

CO₂ full scale: 500 ppm (analog output on channel 2)

CO₂ time constant: 10 sec

CO₂ cell pressure: less than 800 torr

Check zero and span on CO₂ instrument.

5.2.5 SO₂ instrument

SO₂ full scale: 0.5 ppm

Check to see that UV light is flashing.

Check for adequate flow through reaction chamber.

Check for correct vacuum setting.

Check to see that clean filter is installed on instrument inlet.

Check zero and span on SO₂ instrument.

5.2.6 Ozone instrument

Set instrument to sample mode.

Check to see that ozone source is off.

Check to see the clean filter is installed on inlet.

Check zero on O₃ instrument.

5.2.7 FID instrument

Set instrument on 0 -10 ppm range.

Install zero filter on inlet line - set zero on instrument.

Connect 10.5ppm methane gas standard to instrument and adjust span for proper reading.

5.2.8 PID instrument

Set instrument to standby and adjust for zero reading.

Set instrument to 0-20 range.

Connect isobutylene gas standard and adjust meter for correct reading.

5.2.9 Real-time aerosol instrument

Check to see that BNC connector from data acquisition system is connected.

Connect AC battery charger to instrument and plug charger into AC outlet.

Adjust total flow for 2.0 LPM and purge flow for 0.2 LPM.

Position valve in horizontal position for aerosol-free flow to instrument and adjust instrument for zero reading on the 0 - 2 mg/m³ scale.

Depress calibration button to insert scattering device in optical path of the instrument. After instrument stabilities set instrument response at 5.5 mg/m^3 .

Withdraw optical scattering device to return instrument to sampling mode.

Set inlet valve to vertical position to return instrument to aerosol sampling mode.

Full scale range setting 20 mg/m^3

Instrument time constant: 2 sec

5.2.10 Nephelometer

Check nephelometer zeros and adjust as necessary.

Switch instrument to CAL mode and adjust as necessary.

5.2.11 Real-time data acquisition system

On completion of real-time span checks stop data acquisition system.

5.2.12 VOST 1 and 2

Install background VOST cartridge units and filters on VOST 1 and 2 (inside air building). Check and record filter and cartridge numbers for each unit. Check for proper orientation of XAD-2™ and Porapak-R™ resin cartridges.

Measure initial flows on each VOST unit using the dry gas meter and a stop watch. RECORD elapsed time required for 30 L-volume.

5.2.13 SEM/XRF 1

Install leak check background SEM and XRF filter units. Check and record filter numbers.

Measure initial flows on the SEM/XRF filters using the dry gas meter and a stop watch. Record elapsed time required for 30 L volume (XRF) and 5 L volume (SEM).

5.2.14 VOST 3 and 4

Using the bypass mode empty all air from sampling bag.

Install background VOST cartridge units and filters on VOST 3 and 4 (inside airlock). Check and record filter and cartridge numbers for each unit. Check for proper orientation of XAD-2™ and Porapak-R™ resin cartridges.

Switch pumps on momentarily and check for reasonable flow rates through each system.

5.2.15 SEM/XRF 2

Install leak checked background SEM and XRF filters and holders. Record filter numbers in test notebook.

Switch pumps on momentarily and set flow rate for SEM filter to 5 LPM and XRF filter to 25 LPM.

5.3 T-60 to T-45 Background Sample Collection Preparation

Start wide angle video camera.

Set SF₆ release canister and three sets of 32-L tanks.

Collect sample from mixing fan blades.

Turn on mixing fans.

Switch all probe aspirators ON (OAP, FSSP, ASASP).

Check for proper operation of RAM and nephelometer.

Remove 3 inch plug from probe in the air building.

Place explosive charge armed with RP-83 detonator with leads shorted and unconnected to high voltage pulse generator.

5.4 T-45 min Start Background Sample.

Start bubbler samplers record flow reading, RECORD time on. Clear all personnel from air building. Close air-lock door and secure. Switch VOST 1 and 2 and SEM/XRF - RECORD time on. Record building pressure differential and repeat every 5 min.

5.5 T-45 to T-15 Collect Real-Time Data Insure that all real-time instruments are in sampling mode. Set valves for gas sample collection directly from air building. Start HP data acquisition system - collect background gas data for 30 min. Start HP probe data acquisition system - collect background aerosol data for 30 min. Collect 5 DMPS particle samples over a 30 min period - insure that file save mode is ON. Collect 5 APS particle samples over a 30 min period - insure that file save mode is ON.

5.6 T-30 Collect Bag Sample Fill sampling bag. Fill first set of 32-L tanks. Collect 6-L canister sample from mid-level probe. Collect 6-L canister sample from outside building near inflation blower inlet. Fill two 800 ML cans for SF₆ background (floor and ceiling sample).

5.7 T-30 to T-15 Pump Down Bag Sample Metrosonics™ data logger in logging mode. Turn on pumps for VOST 3 and 4 and SEM/XRF 2. Switch CO and CO₂ gas instruments to sampling bag for 3 min. Collect 6-L tank from sampling bag.

5.8 T-15 to T-5 Complete Background Sample/Filter Change-out. Stop HP analog data acquisition.

Stop HP probe data acquisition.

Stop Metrosonics TM logger.

Stop DMPS and APS data acquisition.

Enter air building.

Read final flow on bubblers - read flow - turn off bubblers and change out with new bubblers.

Open SF₆ valve on dump can.

Check and record final flows on VOST 1 and 2 SEM/XRF 1.

Slowly close valves on VOST 1 and 2 - Turn off pumps - Record time off - open valves.

Remove and secure VOST 1 and 2 and SEM/XRF 1 filters and cartridges.

Install new set of cartridges on VOST 1 and 2 and SEM/XRF 1.

Check and record initial flow on VOST 1 and 2 and SEM/XRF 1.

Dump metrosonics data to portable computer (if necessary) - Copy data file to removable disk.

Check metrosonics data file integrity - if OK - clear memory on metrosonics data logger - if not OK repeat file transfer process until satisfactory.

Remove and secure VOST 3 and 4 and SEM/XRF two filters and two cartridges.

Using the bypass mode, empty all air from sampling bag.

Install VOST cartridge units and filters on VOST 3 and 4 (inside airlock). Record filter numbers in notebook.

Switch pumps on momentarily and check for reasonable flow rates through each system.

Install leak-check SEM and XRF filters on bag sampling manifold - Record filter numbers in test notebook.

Switch pumps on momentarily and set flow rate for SEM filter to 5 LPM and XRF filter to 25 LPM.

Final check on all photometrics systems.

Check to see that all probe AC and aspirator lines are plugged in.

Check for proper operation of RAM and nephelometer (Neph on D scale, RAM on 0 - 20 scale).

Insure that all real-time instruments are in sampling mode.

Check to see that ozone instrument is NOT in REMOTE mode. Start HP probe data acquisition system.

Start HP analog data acquisition system.

Check for readiness of 800 Ml cans and 6-L cans for gas sampling.

5.9 Activities Near Time Zero Detonation

5.9.1 T-5

Turn on video camera 1 and 2.

Turn on bubblers RECORD time on.

Evacuate building.

5.9.2 T-1 sec

Start fast action camera.

5.9.3 Detonate TNT

Record time of detonation.

Release SF₆ puff.

Start elapsed timer.

5.10 Post-detonation sampling

5.10.1 T + 2 minutes

Re-enter air lock after "all clear" - NO VEHICLES .

Check for air building integrity - seal major leaks if necessary.

Check that drain plug is still inserted in drain tube.

Switch VOST 1 and 2 and SEM/XRF ON - RECORD time on.

Start DMPS and APS sample collection in continuous mode (APS 120 sec sample - DMPS continuous sample, sampling mode 3).

Begin collecting SF₆ samples every two min (high and low probes).

5.10.2 T + 3 minutes

Flush 4 inch probe prior to collection of sample in bag.

Fill sampling bag - RECORD time in notebook.

Collect sample in second set of 32-L tanks.

Before starting pumps be sure that Metrosonics™ data logger is in logging mode.

Start sampling pumps on sampling bag system.

Switch CO and CO₂ instruments to sampling bag manifold for 3 min.

Collect 6-L canister sample from sampling bag manifold.

Collect 6-L canister outside near inflation blower inlet.

Collect two 6-L can samples from mid-level and ceiling probes.

Replace 4 inch bypass plug.

5.10.3 T+5 minutes

Switch mixing fans ON (3 min).

5.10.4 T+6 minutes

Switch CO/CO₂ back to probe.

5.10.5 T+8 minutes

Switch mixing fans OFF.

Collect 6-L canister sample from mid-level probe.

5.10.6 T+12 minutes

Pump all residual air out of sampling bag.

Change out filters and resin cartridges on VOST samplers 3 and 4 RECORD filter and cartridge numbers in notebook.

Change out SEM/XRF 2 filters and cartridges.

Install second set of VOST cartridges and SEM/XRF filters on bag sampling system.

Metrosonics TM in standby mode.

5.10.7 T+15 minutes

Collect Mixed Sample Flush 4-inch probe prior to collection of sample in bag. Collect sample in bag - RECORD time in notebook. Collect third set of 32-L tank samples. Before starting pumps be sure

that Metrosonics TM data logger is in logging mode. Start sample collection from sampling bag. Collect 6-L canister sample from sampling bag manifold. Collect 6-L canister sample from mid-level probe. Collect 6-L canister sample near inflation blower inlet.

5.10.8 T + 22 minutes

Collect 6-L canister sample from mid-level probe.

5.10.9 T + 30 minutes

Metrosonics TM in logging mode. Fill sampling bag and collect sample on SEM/XRF only. Switch CO/CO₂ instruments to bag sampling manifold (3 min). Collect 6-L canister sample from sampling bag manifold.

5.10.10 T + 33 minutes

Switch CO/CO₂ instruments to probe.

5.10.11 T + 35 minutes

Stop HP-PMS data acquisition system. Stop HP-analog data acquisition system. Stop DMPS and APS. Open door and reenter air building. Collect aerosol sample from fan blade.

Check span on all instruments with data system operating (NO_x-elec, CO₂, CO, FID, PID).

Check and record flows on VOST 1 and 2 - Then shut off samplers using valve closure method.

Check and record flows on SEM/XRF 1.

After flow checks remove and secure VOST cartridges and filters both inside the air building and in the airlock.

Backup files to floppy disks (HP-Analog, HP-PMS, DMPS, APS) - check to see that disk are labeled correctly.

Dump Metrosonics TM to portable computer - check for file integrity before clearing memory on the Metrosonics TM.

ANDRULIS RESEARCH

LOI 1 DATA REDUCTION AND ANALYSIS

Outline of Procedure:

1. Data Receipt.

- a. Individual sample assay (wt/vol) with all blanks and inserted standards.
- b. Calibration curve - with all data used to generate the best fit line.
- c. Algorithm with constants that are used in computing engineering units at standard conditions (SC)(wt/vol) from the measured units; e.g. volts to wt/vol.
- d. Real-time data recorded in 5-sec intervals for all indirect sampling and for direct sampling recorded in 1-sec intervals.

2. Data Analysis.

a. Leak Rate Trials (on site)

- (1) Visually inspect graphic output of trial (elapsed time versus concentration).
- (2) Compute slope (as required for on-site decision making).

time samples.

b. Homogeneity Trials (on site)

- (1) Visually inspect graphic output of trial (elapsed time versus concentration) for all real-

- (2) Compare expected concentration of SF_6 with actual measured concentration of SF_6 .
- (3) Perform additional computations that may be needed for on-site decision making.

c. Trinitrotoluene Trials

- (1) Examine real-time graphic output after each trial to determine if the trial was a success (based upon slope of response curve, and initial concentration of constituents).
- (2) Check calibration curves and assure that standards concentration spans the range of the sampled compounds concentration.
- (3) Verify at least one non-zero value (measured units) with the algorithm used for converting to engineering units.
- (4) Correct all concentrations for measured background as required.
- (5) Calculate the total mass of each elemental and chemical compound assayed, using the volume of the BangBox (BB) and the extrapolated estimate of concentration at detonation time.
- (6) Calculate the emission factor using the carbon balance procedure for each measured emission. Using these emission factors from each sampling point, determine the total mass of each elemental and chemical compound. (Details of this method have previously been presented by Sandia National Laboratories and approved by the Open Burning/Open Detonation (OB/OD) Technical Steering Committee as a method for determining the mass of the emissions from OB/OD.)
- (7) Compare the results from (5) and (6) above to determine the equality of the following:
 - (a) 32-L sampling system results with indirect 1-m^3 bag results
 - (b) non-homogeneous aerosol emission factors with homogeneous aerosol emission factors

(8) Compare the assay concentrations results from the supercritical Fluid chromatograph/mass spectrometer with the results from the gas chromatograph/mass spectrometer method. The comparison will use the data and the U.S. Environmental Protection Agency (EPA) report from samples spiked by EPA, the concentration values from the BB, and the laboratory standards inserted within the assay of the BB samples. Additionally, the detection limit and the variability of each method will be examined.

(9) Compare the concentration of CO and CO₂ from the 6-L canister with the concentration for the real-time fixed-wing-aircraft system.

(10) All results from the analysis/comparison will be furnished the Program Manager and only upon his approval will additional distribution be made.

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OREGON GRADUATE CENTER

LOI 1 VOC Collection Analysis System

Adapted from EPA Compendium Method TO-14

R. A. Rasmussen

1.1 Canister Construction

- 1.1.1 Type 304 stainless steel with 20 gage wall thickness used to fabricate stainless steel air sampling canisters. All welded seams welded metal-to-metal (no welding rod used) under Argon shield for T.I.G. weld, 100% penetration.
- 1.1.2 Sample sizes available: 850 mL, 3.2L, 6L, 15L, and 32L. For simultaneous paired air sampling 3.2-L canisters are used.
- 1.1.3 All internal surfaces are electropolished using Moleetrics, Inc. (Carson, CA), Type Power Kleen™ solution, commonly referred to as the SUMMAR process. This treatment leaves an enriched surface layer of chrome-nickel oxide that is designated as "passivated." After electropolishing, said surfaces are thoroughly washed with DI water and cleaned of any residual organics by a vacuum bake-out at 120 °C to 100 mTorr for several hours.
- 1.1.4 Valve configurations are typically a single Nupro™ SS-4H4 bellows stem valve. To ensure leak-tight construction to air sampling manifolds, the valves are fitted with Cajon™ VCRR 1/4-inch male connectors. These fittings use an expendable metal disc to provide vacuum-tight connections. Swagelok™ fittings are not acceptable for multiple use leaktight vacuum assemblies.

1.2 Canister Integrity

1.2.1 All canisters are hydrostatically tested to 20 Atm. (300 psig).

1.2.2 Recommended operational pressure range is -30 inches Hg to 2 Atm. (30 psig).

1.2.3 All containers are helium leak-tested to 1×10^{-9} cc/sec.

1.3 Canister Cleaning System

1.3.1 Vacuum pump (Alcatel, Hingham, MA, Model M2008A). Capable of evacuating sample canister(s) to an absolute pressure of < 0.05 mm Hg (50 mTorr).

1.3.2 Manifold - stainless steel manifold with connections for simultaneously cleaning two-four canisters.

1.3.3 Shut-off valve(s) - five on-off Nupro™ SS4H4 valves.

1.3.4 Stainless steel vacuum gage (Varian, Palo Alto, CA, Model VH3) capable of measuring vacuum in the manifold to an absolute pressure of 0.05 mm Hg or less.

1.3.5 Cryogenic trap (2 required) - all glass, standard mechanical roughing pump type open tubular trap cooled with liquid nitrogen (-196°C) to prevent contamination from back diffusion of oil from vacuum pump.

1.3.6 Stainless steel pressure gauges (2) (Span Instruments, Plano, TX) all SS, 0-345 kPa (0-50) psig) to monitor zero air pressure.

1.3.7 Stainless steel flow control valve, Nupro™ SS-4H4 - to regulate flow of zero air into canister(s).

1.3.8 Humidifier - pressurizable glass water bubbler containing high performance liquid chromatography (HPLC) grade deionized water or other system capable of providing

moisture to the zero air supply.

- 1.3.9 Isothermal oven for heating canisters (0-150 °C), 16 x 24-inch I.D., special laboratory construction. Note: Very important that Nupro™ SS-4H4 valves remain outside of oven during bake-out procedure.

1.4 Calibration System and Manifold.

- 1.4.1 Calibration manifold - SS manifold, SS tubing internally passivated or electropolished. Sampling ports and internal baffles for flow disturbance to ensure proper mixing. System is designed to handle pressures up to 350 psig. Two-stage high purity regulators (Veriflow, Richmond, CT, Model IR501B-4-SSR) are used to reduce pressures from EPA audit cylinders.

- 1.4.2 Humidifier - 500-mL impinger flask containing HPLC grade deionized water.

- 1.4.3 Electronic mass flow controllers - ranges 0 to 5 L/min, 0 to 50 cm³/min (Tylan Corporation, Carson, CA, Model FC260, or equivalent).

- 1.4.4 Teflon™ or SS in-line filter(s) for particulate control - 47-mm Teflon™ or 10-m-nominal SS discs, best source.

2. Standards, Reagents, and Materials

- 2.1 Gas cylinders of helium, hydrogen, nitrogen, argon/methane (5/95%), and zero air - ultrahigh purity grade, best source.

- 2.2 Gas calibration standards - cylinder(s) containing the following compounds of interest are available:

vinyl chloride	1,2-dibromoethane
vinylidene chloride	tetrachloroethylene

1,1,2-trichloro-1,2,2-	chlorobenzene
trifluoroethane	benzyl chloride
chloroform	hexachloro-1,3-butadiene
1,2-dichloroethane	methyl chloroform
benzene	carbon tetrachloride
toluene	trichloroethylene
FreonR 12	cis-1,3-dichloropropene
methyl chloride	trans-1,3-dichloropropene
1,2-dichloro-1,1,2,2-tetra-	ethylbenzene
fluoroethane	o-xylene
methyl bromide	m-xylene
ethyl chloride	p-xylene
FreonR 11	styrene
dichloromethane	1,1,2,2-tetrachloroethane
1,1-dichloroethane	1,3,5-trimethylbenzene
cis-1,2-dichloroethylene	1,2,4-trimethylbenzene
1,2-dichloropropane	m-dichlorobenzene
1,1,2-trichloroethane	o-dichlorobenzene
	p-dichlorobenzene
	1,2,4-trichlorobenzene

2.3 Primary reference standards are traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to an NBS/EPA-approved Certified Reference Material (CRM). For hydrocarbon speciation we use two different NBS SRM's: Benzene in N₂, #1805 at 0.25 ppm; Propane in N₂, #1665b at 3 ppm; Methane in Air, #1658a at 1 ppm and #1659 at 10 ppm. For carbon monoxide we use NBS SRM CO in Air #2612 at 10 ppm.

2.4 Gas purifiers - connected in-line between hydrogen, nitrogen, and zero air gas cylinders and system inlet line, to remove moisture and organic impurities from gas streams (built to own specifications using mol-sieve).

2.5 Deionized water -high performance liquid chromatography (HPLC) grade, ultrahigh purity (for humidifier), best source.

2.6 Hexane - for cleaning sampling system components, reagent grade, best source.

2.7 Methanol - for cleaning sampling system components, reagent grade, best source.

3. Sampling System

3.1 System Description

3.1.1 Pressurized Sampling (with Teflon™ diaphragm-type pump).

3.1.1.1 Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control device to achieve a typical 10-30 psig final canister pressure.

3.1.1.2 In pressurized canister sampling the pump (Model FC1121, BRC, Hillsboro, OR) draws in ambient air from the sampling manifold to fill and pressurize the sample canister.

3.1.2 All Samplers

3.1.2.1 A flow control device (Veriflow, Richmond, CA, Model SS-423 OGC) is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled to about one atmosphere above ambient pressure over the desired sample period. For example, if a pair of 3.2L canisters are to be filled to 2 atmospheres (15 psig) absolute pressure in 3 hours, the flow rate can be calculated by:

Equation (1) Flow Rate Calculation

$$F = 2 \times \frac{6400}{3 \times 60} = 71 \text{ cm/min}$$

- 3.1.2.2 For automatic operation, the timer (Chronotrol, San Diego, CA, Model CD-4) is wired to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and close the valve when stopping the pump.
- 3.1.2.3 The use of the Skinner (Columbus, OH) Magnelatch™ (Model VTR 1211) valve avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton™ valve seat material. The Magnelatch™ valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods.
- 3.1.2.4 The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period.
- 3.1.2.5 Prior to field use, each sampling system has passed a humid zero air certification (see TO-14, Section 12.2.2). All plumbing has been checked carefully for leaks. The canisters also have passed a humid zero air certification before use (see EPA-TO-14, Section 12.1).

3.2 Sampling Procedure

3.2.1 The sample canisters have been cleaned and tested according to the procedure in EPA-TO-14, Section 12.1.

3.2.2 The sample collection system is assembled and has met certification requirements as outlined in EPA-TO-14, Section 12.2.3. [Note: The sampling system should be contained in an appropriate enclosure if placed out-of-doors.]

3.2.3 To verify correct sample flow "practice" canisters are used in the sampling system. For the pump-driven system the practice canisters are not opened as the flow is measured at the outlet of the system. A mass flow meter is attached to the outlet line of the sampler and the vent. The valve is opened. The sampler pump is turned on and the reading of the mass flow meter or calibrated rotameter is compared to the flow rate specified: 71 mL/min. The valves should agree with +10%. If not, the sampler flow controller needs to be set to the desired setting.

3.2.4 The sampler is turned off. Note: Any time the sampler is turned off, wait at least 30 seconds to turn the sampler back on.

3.2.5 See attached (Appendix A) detailed operating instructions for R. A. Rasmussen Integrated Air Sampling System.

3.2.6 An identification tag is attached to the canister. Canister serial number, sample number, location, date, time, and comments are recorded on the tag.

4. Analytical System

4.1 System Description

4.1.1 GC-FID System

- 4.1.1.1 The analytical system is composed of a gas chromatograph (HP5790, Avondale, PA) equipped with a capillary column and a flame ionization detector. In typical operation, sample air from pressurized canisters is vented to the analytical system from the canister at a flow rate of 80 cm³/min. For analysis 500 cm³/min of sample gas is used. Sub-ambient pressure canisters are connected directly to the inlet. The gas volume is measured via the increase in pressure in the fixed volume (4-L) vacuum flask. The sample gas stream is routed through a six-port chromatographic valve (Carle or Valco) and into the cryogenic trap. [Note: This represents a 6.2-minute sampling period at a rate of 80 cm³/min.] The trap is 1/8-inch OD x 8 inches packed with 60-80 mesh glass beads and is cooled to -183 °C by immersion in LOX cryogen. VOCs are condensed on the trap surface while N₂, O₂, and other sample components are passed to the vacuum reservoir. After the organic compounds are concentrated, the valve is switched and the trap is heated. The re-volatilized compounds are transported by helium carrier gas at a rate of 4 cm³/min to the head of a wide bore DB-1 capillary column (0.32 mm x 60 m). Since the column initial temperature is at -60 °C, the VOCs are cryofocussed on the head of the column. The oven temperature is programmed from -60 to 150 °C at 4 °C/min after an initial 2-minute hold. The VOCs in the carrier gas are chromatographically separated. The FID detector senses the presence of the carbon in the speciated VOCs, and the response is recorded by either a strip chart recorder or an electronic integrator.
- 4.1.1.2 Helium is used as the carrier gas (3 cm³/min) to purge residual air from the trap at the end of the sampling phase and to carry the re-volatilized VOC through the wide GC column. Moisture and organic impurities are removed from the helium gas stream by a chemical purifier installed in the GC.
- 4.1.1.3 Gas scrubbers containing Drierite™ or silica gel and 5A molecular sieve are used to remove moisture and organic impurities from the zero air, hydrogen, and nitrogen gas streams. [Note: The purity of gas purifiers is checked prior to use.]
- 4.1.1.4 All lines should be kept as short as practical. All tubing used for the system

should be chromatographic grade stainless steel connected with stainless steel fittings.

4.1.1.5 The FID burner air, hydrogen, nitrogen (make-up), and helium (carrier) flow rates are set to obtain an optimal FID response while maintaining a stable flame throughout the analysis. Typical flow rates are: burner air, 450 cm³/min; hydrogen, 30 cm³/min; nitrogen, 30 cm³/min; helium, 3 cm³/min.

4.2 GC-FID Calibration

4.2.1 At the beginning of each day three analyses of a single point working standard (neohexane) are made. If the results fall within +2%, the average of the response is used to calibrate the successive analyses. If the values for the three initial neohexane analyses exceed +2%, a fourth and/or fifth analysis is performed. Neohexane as the daily calibration standard has been used in our laboratory for 14 years. The present values are directly traceable to our primary NBS SRM benzene and propane standards. The working standards are metered with and without humidified zero air, depending upon the analyses.

4.2.2 As an alternative, a multipoint humid static calibration (three levels plus zero humid air) is sometimes performed on the GC-FID system. During the humid static calibration analyses, three SUMMAR passivated canisters are filled each at a different concentration between 1 and 20 ppbv from the calibration manifold using a mass flow control arrangement. These calibration standards are each analyzed twice. The expected retention times are used to verify proper operation of the GC-FID system. A calibration response factor is determined for each analyte and used where appropriate. The computer calibration table is updated with this information.

4.2.3 Routine Calibration: The GC-FID system is calibrated daily with a one-point calibration. The system is calibrated either with the single point neohexane standard in a high pressure (2000 psig) tank procedure or with a low pressure (<40 psig) 6L SUMMAR passivated canister filled with humid calibration standards. After the single

point calibration, the GC-FID analytical system is challenged with a humidified zero gas stream to ensure that the analytical system returns to specification (less than 0.2 ppbv of selective organics).

4.3 GC-FID System Performance Criteria

4.3.1 Humid Zero Air Certification

4.3.1.1 Before system calibration and sample analysis, the GC-FID analytical system is optimized.

4.3.1.2 Periodically the GC-FID system is challenged with humid zero air.

4.3.1.3 Analytical systems contaminated with less than 0.2 ppbv of targeted VOCs are acceptable.

4.3.2 GC Retention Time Windows Determination.

4.3.2.1 For proper identification, the retention time windows must be established for each analyte.

4.3.2.2 To do this properly, the GC system must be within optimum operating conditions.

4.3.2.3 Three injections of a diluted auto exhaust standard containing all compounds for retention time window determination are made. [Note: The retention time window must be re-established for each analyte periodically or when drift in the R.T. is observed.]

4.3.2.4 The standard deviation of the three absolute retention times for each single component standard is calculated. The retention window is defined as the mean plus or minus three times the standard deviation of the individual retention times for each standard.

4.3.2.5 The retention time windows for each standard are determined on each GC column whenever a new GC column is installed or when major components of the GC are changed. The data are noted and retained in a notebook in the laboratory as part of user SOP and as a quality assurance check of the analytical system.

4.4 Analytical Procedures

4.4.1 Canister Receipt

4.4.1.1 The overall condition of each sample canister is observed. Each canister is expected to be received with an attached sample identification tag.

4.4.1.2 Each canister is recorded in the logbook. Noted on the identification tag are the date received and the initials of the recipient.

4.4.1.3 The pressure of the canister is checked by attaching a pressure gauge to the canister inlet. The canister valve is opened briefly and the pressure (kPa, psig) is recorded. Final cylinder pressure is recorded on the canister sampling field data sheet.

4.5 GC-FID Analysis

4.5.1 The analytical system should be humid zero air certified and calibrated through working standards directly referenced to NBS SRMs.

4.5.2 Sixty minutes are required for each sample analysis: 15 minutes for system initialization and sample collection, 40 minutes for analysis, and 5 minutes for post-time, during which a report is printed.

4.5.3 The helium and sample mass flow controllers are checked and adjusted to provide correct flow rates for the system. Helium is used to purge residual air from the trap

at the end of the sampling phase and to carry the re-volatilized VOCs from the trap onto the GC column and into the FID. The hydrogen, burner air, and nitrogen flow rates are also checked. The cryogenic trap is connected and verified to be operating properly while flowing cryogen through the system.

4.5.4 The sample canister is connected to the inlet of the GC-FID analytical system. The canister valve is opened and the canister flow is vented to flush the system prior to passing the sample through the freezeout loop into the receiving vacuum-volume reservoir. The VOCs are condensed in the trap.

4.5.5 The six-port valve is switched to the inject position, and the canister valve is closed.

4.5.6 The electronic integrator is started.

4.5.7 After the sample is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -60 °C, the VOCs are cryofocussed on the column. Then the oven temperature (programmed) increases and the VOCs elute from the column to the FID assembly.

4.5.8 The peaks eluting from the detectors are identified by retention time while peak areas are recorded in area counts.

4.5.9 The response factors are multiplied by the area counts for each peak to calculate mg/m^3 for the unknown sample.

4.5.10 Each canister is analyzed once. [Note: paired samples are collected and an equal aliquot from each is used in the analyses.]

5. Cleaning and Certification Program

5.1 Canister Cleaning and Certification

- 5.1.1 All canisters must be clean and free of any contaminants before sample collection.
- 5.1.2 All canisters are leak tested by pressurizing them to approximately 30 psig with zero air. The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If leak tight, the pressure should not vary more than +2 psig over the 24-hour period. Alternately the canisters are tested with a He-leak MS system to 1×10^{-9} cc/sec.
- 5.1.3 A canister cleaning system is generally used to facilitate recycling of the cans. A cryogen (LN₂) is added to the vacuum and zero air supply traps. The canister(s) are connected to a 2-position manifold. The manifold-vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The valve to the vacuum pump is opened after the manifold vent shut-off valve is closed. The canister(s) are evacuated to 100 mTorr for at least one hour.
- 5.1.4 The vacuum line is shut off and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 30 psig. A Bryon Instruments Model 25 (Raleigh, NC) zero gas generator system is used; the flow rate is limited to maintain optimum zero air quality.
- 5.1.5 At the end of cycle #1 the zero air shut-off valve is closed, and the canister(s) are allowed to vent down to atmospheric pressure through the manifold vent shut-off valve. The vent shut-off valve is closed. Steps 5.1.3 through 5.1.5 are repeated two additional times for a total of three evacuation/pressurization cycles for each set of canisters.
- 5.1.6 At the end of the evacuation/pressurization cycle, the canister is pressurized to 30 psig with humid zero air. The canister is then analyzed by a GC-FID or ECD analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) are not used.
- 5.1.7 The canister is reattached to the cleaning manifold and is then re-evacuated to <50 mTorr and remains in this condition until used. The canister valve is closed. The

canister is removed from the cleaning system, and the canister connection is capped with a brass Swagelok™ fitting. The canister is now ready for collection of an air sample. An identification tag is attached to the neck of each canister for field notes and chain-of-custody purposes.

5.1.8 As an option to the humid zero air cleaning procedures, the canisters are heated in an isothermal oven to 100 °C during Section 11.1.3 to ensure that lower molecular weight compounds (C2-C8) are not retained on the walls of the canister. For sampling heavier, more complex VOC mixture, the canisters should be heated to 120 °C. Once heated, the canisters are evacuated to 50 mTorr. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by the GC-FID system. Any canister that has not tested clean (less than 0.2 ppbv of targeted compounds) are not used. Once tested clean, the canisters are re-evacuated to 50 mTorr and remain in the evacuated state until used. [Note: The Nupro SS-4H4 valves must be positioned outside of the oven; only the canister body is heated; otherwise, severe damage to the valve may occur.]

5.2 Sampling System Cleaning and Certification

5.2.1 Cleaning Sampling System Components

5.2.1.1 Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50 °C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent only when needed.

5.2.1.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100 °C for 12 to 24 hours.

5.2.1.3 Once the sampler is assembled, the entire system is purged with humid zero air

for 24 hours.

5.2.2 Humid Zero Air Certification

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv of targeted compounds) have occurred when challenged with the test gas stream.]

5.2.2.1 The cleanliness of the sampling system is determined by testing the sampler with humid zero air.

5.2.2.2 The sampler is connected to the manifold and the humid zero air is passed through the system.

5.2.2.3 The humid zero gas stream passes through the sampling system to a GC-FID analytical system at 80 cm³/min so that a 500 cm³/min is pulled through the six-port valve and into the cryogenic trap. After the sample (500 mL) is reconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -60 °C, the VOCs are cryofocussed on the column. Then the oven temperature (programmed) increases and the VOCs begin to elute and are detected by the GC-FID. The analytical system should not detect greater than 0.2 ppbv of targeted VOCs in order for the sampling system to pass the humid zero air certification test.

6. Performance Criteria and Quality Assurance

6.1 Standard Operating Procedures (SOPs)

6.1.1 The SOPs given in Sections 1-5 have described the following activities: (1)

Manufacture, safety factor, assembly, calibration, leak check, and operation of specific sampling systems, and equipment used; (2) preparation, storage, shipment, and handling of samples; (3) assembly, leak-check, calibration, and operation of the analytical system for the specific equipment used; (4) canister storage and cleaning; and (5) data recording and processing.

6.1.2 Specific stepwise instructions have been provided in the SOPs and are available to and understood by the laboratory personnel conducting the work.

6.2 Method Relative Accuracy and Linearity

6.2.1 Accuracy is determined by measuring VOC standards from an NBS or EPA audit cylinder into a sampler. The contents are then analyzed for the components contained in the audit canister. Percent relative accuracy is calculated:

$$\% \text{ Relative Accuracy} = \frac{X - Y}{X} \times 100$$

Where: Y = concentration of the targeted compound recovered from sampler.

X = concentration of VOC targeted compound in the NBS-SRM or EPA-CRM audit cylinders.

6.2.2 If the relative accuracy does not fall between 90 and 110 percent, the field sampler is not used. Historically, concentrations of the C2-C9 hydrocarbons are accurately measured with the FID. The detector is very linear over concentration ranges of 104 or more. All of the software for the Hewlett-Packard series of GC 5790's with 3390A data processors, GC 5890's with 3393A or 3396 recorder-integrators are designed to accommodate multilevel calibration entries, so the correct response factors are automatically calculated as well as concentrations in the range of ambient air analysis.

6.3 Quality Assurance

6.3.1 Sampling System

- 6.3.1.1 Pre- and post-sampling measurements with a mass flow meter or rotameter for flow verification of sampling system should be made periodically in the field.
- 6.3.1.2 Canisters are pressure tested to 30 psig +2 psig over a period of 24 hours or preferably He leak-tested to 1×10^{-9} He cc/sec.
- 6.3.1.3 All canisters are certified clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.
- 6.3.1.4 All field sampling systems are certified initially clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.
- 6.3.1.5 All field sampling systems have passed an initial humidified calibration gas certification [at VOC concentrations levels expected in the field (e.g., 0.5 to ppbv)] with a recovery of greater than 90%.

6.4.1 GC-FID System Performance Criteria

- 6.4.1.1 The GC-FID analytical system, prior to analysis, is certified to be clean (less than 0.2 ppbv of targeted VOCs) through a humid zero air certification.
- 6.4.1.2 The GC-FID analytical system retention time windows for each analyte prior to sample analysis are verified when a new GC column or major components of the GC system are altered since the previous determination.
- 6.4.1.3 All calibration gases are traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM).

6.4.1.4 The retention time windows are re-established continuously throughout the course of the analytical period.

6.4.1.5 The long-term quality control results for measuring our neohexane working standard of 226 ppbv against two different NBS SRMs are given in Table 1. The precision of these intensive sets of analyses (usually 3 days are required to complete the tests) are very good: 1-4%. The data have been systematically obtained since 1985. Currently we intercalibrate our working standard against two NBS SRMs, benzene and propane, every six months. No difference or bias is observed for either NBS SRM used to calibrate the neohexane standard. The data suggest that the neohexane standard is very stable and is accurately referenced to the NBS SRMs. The NBS-SRMs concentrations and hydrocarbon species are very different from one another: 2.87 vs. 0.254 ppmv for propane and benzene respectively. This supports our contention that our neohexane standard is accurately referenced to the NBS-SRMs. We have been using neohexane as our daily working standard for 14 years. The prime reason it was selected is that it is one of the few hydrocarbons that can be used as an internal standard in an urban ambient air sample because it is not a product of auto exhaust. The lower detection limit of our GC-FID system using 500-mL air samples is 0.2 mg/m^3 for benzene. The precision of analysis for benzene at 3.0 mg/m^3 is +10% (2s). In clean air along the California coast observed benzene levels are 0.3 to 3 mg/m^3 , whereas at dirty sites 3-12 mg/m^3 values are measured. The lower detection limit

Table 1 Quality Control Results

Primary Standards	Neohexane Working Standards			Percent	
	Given Value	Analysis	Assigned	Measured	Difference
	ppmv	Date		ppmv	ppmv
Benzene NBS-SR #1805 CAL 5679	0.254	Dec 1985	0.226	0.224	2
Benzene NBS-SR #1805 CAL 5679	0.254	Jun 1987	0.226	0.233	4
Benzene NBS-SR #1805 CAL 5679	0.254	Nov 1987	0.226	0.219	-3
Benzene NBS-SR #1805 CAL 5679	0.254	Apr 1988	0.226	0.221	-3
Propane NBS-SR #1665-B FF27623	2.87	Mar 1988	0.226	0.218	4
Benzene NBS-SRM #1805 CAL 5679	0.254	Oct 1988	0.226	0.222	± 2
Propane NBS-SR #1665-B FF27623	2.87	Oct 1988	0.226	0.224	± 1
Benzene NBS-SRM #1805 CAL 5679	0.254	Jun 1989	0.226	0.205	-9
Propane NBS-SRM #1665-B FF27623	2.87	Jun 1989	0.226	0.225	± 1
Benzene NBS-SR #1805 CAL 5679	0.254	Oct 1989	0.226	0.226	0
Propane NBS-SRM #1665-B FF27623	2.87	Oct 1989	0.226	0.227	0
Benzene NBS-SRM #1805 CAL 5679	0.254	Apr 1990	0.226	0.226	0
Propane NBS-SRM #1665-B FF27623	2.87	Apr 1990	0.226	0.226	0

of our GC-FID system of 0.1 to 0.2 mg/m³ per compound for a 500-mL sample is determined more by the threshold limits of the HP electronic integrators than by the physical discernment of a peak. The lower detection limits for propane (C3), neohexane (C6), and benzene (C6 aromatic) are consistent within the analytical parameters used. Therefore, we believe that from C3 to C9 the lower detection limit for identified and unidentified hydrocarbons species is essentially the same: ~0.2mg/m³.

- 6.4.1.6 The absolute accuracy of our calibration standard is determined against the two NBS-SRM standards. The measured value for 7 determinations over three years is 0.224 ppmv +0.005, or +2.3%. This suggests that our neohexane standard is less than its assigned value of 0.226 ppmv by 0.002 ppm, or 0.9%. We do not believe this is a real difference.

ENVIRONMENTAL LABS, INC.

LOI 1 PROCEDURES FOR WRITING LETTERS OF INSTRUCTION (LOI)

1. Purpose

QA/QC practices require that all routine activities having an impact on data quality be fully documented. These day-to-day routine practices should be itemized in a clear, explicit, somewhat detailed, step-by-step manner so that most individuals could follow them in a uniform and consistent manner.

2. Scope

LOIs should be prepared for all routine activities associated with the OB/OD project. Such activities may include:

- Field or Laboratory Sampling
- Field or Laboratory Analysis
- Instrument or Method Calibrations
- Preventative and Corrective Maintenance
- Internal QC Procedures
- Site Selection
- Sample Preparation and Storage
- Preparation and use of Spiked Samples
- Instrument/Equipment Selection and use
- Determination of Detection Limits and Limits of Quantification
- Sample Handling and Transportation
- Conduct Performance Audits
- Data Reduction and Analysis

For the "Bang-Box" portion of the OB/OD project, LOI can be more general than SOP.

Once the SOP are written, they will be reviewed for adequacy by the QA Officer and Technical Coordinator, and kept in each Laboratory's files and made available during QA visits. LOI may also be changed or revised during the study to conform with the actual work effort. However, these changes should be clearly documented and the old LOI replaced with the new one (see document control section below).

3. Format

The format and outline of LOI may vary somewhat depending on the activity. However, all LOI should contain the following:

Title

Outline of procedures

References (if appropriate)

Document control block in upper right hand corner

Signature line for project officer and signature line for QA

4. Document Control

Each page of the LOI should contain a document control block in the upper right corner containing: LOI number, date of issue, revision number, and page number similar to that used for this document.

5. Where to Send LOI

A copy of each LOI should be kept in the originating laboratory and a signed copy should be sent to:

Dr. Gary M. Booth
Environmental Labs, Inc.
1125 South 550 East
Springville, Utah 84663

All LOI should be received by Nov 28, 1988. If you have any questions, you may call Gary Booth at (801) 378-2458.

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LAWRENCE-BERKELEY LABORATORY

LOI 1 LOI FOR METALS ANALYSIS BY X-RAY FLUORESCENCE ANALYSIS

This report was submitted in support of the Preliminary Test, Open Burning/Open Detonation Study, by Robert D. Giauque, Joseph M. Jaklevic, and Linda Sindelar of the Lawrence Berkeley Laboratory, University of California, Berkeley, California.

1. Introduction.

This report describes data acquisition and analysis procedures used at Lawrence Berkeley Laboratory (LBL) for the x-ray fluorescence (XRF) analysis of aerosol filter specimens. The analysis was obtained using an LBL-designed and built x-ray fluorescence system using an energy dispersive x-ray detector with pulsed excitation obtained from multiple secondary targets. The system had been developed under a previous interagency agreement with the EPA and was specifically designed for the automated analysis of filters acquired from dichotomous samplers. The LBL project manager was J.M. Jaklevic; the person responsible for the sample handling and data analysis is R. D. Giauque.

2. Specimen Reception and Documentation

a. Filters were received at LBL in plastic 47-mm petri slides. The specimens were stored in their shipping bags and slide trays at all times, except during the XRF analysis period.

b. Included with each set of filter specimens was a shipping list giving sample identification information.

c. Upon receipt of the samples, the data was recorded in a log book, along with the sample site, sample identification group, and the name of the study program for which the specimens were collected.

3. Setup of the X-Ray Spectrometer Semiconductor Detector Amplifier

a. Prior to the analysis of the aerosol specimen in an individual slide tray, the setup of the bias cut and the gain of the amplifier for the semiconductor was monitored and adjusted slightly, if necessary. The setup was achieved using our standard filter that contains S, Cu. and Ag.

b. The standard filter was prepared by aspirating dilute solutions containing $(\text{NH}_4)_2\text{SO}_4$, $\text{Cu}(\text{NO}_3)_2$, and AgNO_3 into a dichotomous aerosol sampler. The filter from the fine particle size fraction was used as the standard. Our standard contained $22.1 \mu\text{g}/\text{cm}^2$ of Cu, and $13.9 \mu\text{g}/\text{cm}^2$ of Ag. Each of these three elements served as the standard for a separate x-ray excitation condition. In each of these conditions a specific secondary target (fluorescence) exposed to radiation from a tungsten x-ray tube. The x-ray tube was operated at a present voltage that yields optimum signal-to-background ratios for a given group of elements to be determined.

c. For our determinations, the fluorescers employed were Ti, Mo, and Sm. The x-ray tube voltages employed for these targets were 50, 60 and 74 kV, respectively. The standard elements for each of these excitation conditions were S for Ti, Cu for Mo, and Ag for Sm, respectively. The entire x-ray spectrometer system has been previously described elsewhere.

d. The setup of the bias cut and the fine gain of the amplifier was achieved by verifying that a low energy x-ray peak (S K-alpha at 2.31 keV) and that a high energy x-ray peak (Ag K alpha at 22.10 kev) was centered in the preset channel windows used for our analysis program.

e. First, using the Ti fluorescer, a spectrum was acquired for the standard. If the S peak was not centered between channels 48 and 58, the bias cut was adjusted and a new spectrum was acquired. The process was repeated until the S peak was properly centered in the preset channel window. Then, using the Sm fluorescer, a spectrum was acquired for the standard. If the Ag peak was not centered between channels 658 and 678, the fine gain was adjusted and a new spectrum was acquired for the standard. The process was repeated until the Ag peak was properly centered in the preset channel window. Using the Ti fluorescer, the above setup was repeated for the S peak. If a

bias cut adjustment was required, then the process of using the Sm fluorescer and adjusting the fine gain to center the Ag peak, in the preset window was repeated. The setup procedure typically took between 5 and 30 min. The x-ray spectrometer system was in a room that was air conditioned in order to maintain electronic stability.

4. Acquisition of X-Ray Spectra for Element Determinations

a. Some of the slide frames fit tightly in the slide trays used for shipping the dichotomous aerosol specimens. These frames will not feed into our x-ray spectrometer with our existing mechanical sample-feeding system. Consequently, prior to analysis, the slides for an individual tray were removed and placed in sequential order in an LBL linear slide-frame box that does not bind the sample frames. Care was taken to ensure that the sample order was maintained and that the filter deposits are not disturbed in any manner.

b. The computer used to operate the LBL x-ray spectrometer and to generate the input and the output data was an IBM, PC. All of the programs that were used were established at LBL and are written in the BASIC Programming language. The computer system has two floppy disk drives and one removable 5-megabyte hard disk drive. The computer has 610 kilobytes of memory.

c. Three x-ray spectra (one for each excitation condition) were acquired for each aerosol specimen. This was accomplished using our LBL ANAL program that directed the mechanical operation of the x-ray spectrometer and wrote the 1024 channels of spectrum data on a 5 megabyte removable hard disk. Each spectrum on a disk had a specific sequential number, and contains identification information, date and time that the spectrum acquisition was concluded, slide position number, fluorescer, and the number of beam dumps. For our determinations the x-ray tube pulses for 20,000 beam dumps for each of the three excitation conditions. All spectra were acquired with the specimen purged in a helium atmosphere. When compared with air the helium atmosphere greatly minimized the spectral background due to scattered excitation radiation. Additionally, the helium atmosphere substantially reduced the absorption of low energy x-rays.

d. Each day that aerosol specimens were analyzed, spectra were acquired for the standard filter, a control filter, and a blank filter. The standard filter was used to normalize for any small

day-to-day variation in the output intensity of the x-ray tube. The control filter contained 19 elements. The 19 elements are S, K, Ca, Ti, Mn, Fe, Ni, Cu, Zn, Rb, Sr, Zr, Mo, Ag, Cd, Sn, Sb, Ba, and Pb. The results ascertained for the control filter were used to verify the stability of the spectrometer system and to substantiate the consistency of the analysis program. The ratio of the determined-to-established values for each of the 19 elements in the control filter were plotted. Any deviations beyond those expected due to statistical considerations were evaluated. Those found to be unreasonable (greater than three sigma) normally led to reanalysis of the dichotomous aerosol specimens analyzed on that specific day. It has been our experience over the past 2 years of analysis that the deviations were always within statistical limits, except for those occasions when major malfunctions of the system have occurred.

5. Calculation of Initial Element Concentration Values for Each Filter

a. Using our RECDATA program, a RECDATA file was established that contains the sample identification, sequential file number, fluorescer number, the background program number, and the standard spectrum number for each filter spectrum stored on the 5-megabyte removable hard disk. For this input, a sequential RECDATA file was assigned for each of the records in the RECDATA file. The RECDATA file was written on a floppy disk.

b. The XRF program was used to merge the information from the RECDATA file with the spectral data stored on the hard disk. For each sample spectrum, the total counts in the fixed channel windows assigned to each element were integrated. These windows encompassed between 80 and 90 percent of the total peak of the x-ray line chosen for analysis. For all elements except Pb, the K-alpha x-ray line was used. For Pb, the L-beta x-ray line was utilized. Additionally, a fixed channel window for the intense region of the backscattered excitation radiation was integrated. For the Ti fluorescer, the fixed element channel windows were integrated for Al, Si, Cl, K, and Ca. Similarly, for the Mo fluorescer, integrations were performed for Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, and Pb. For the Sm fluorescer, integrations performed include Zr, Mo, Ag, Cd, Sn, I, and Ba.

c. A blank filter was included with each of the tray filters. The x-ray spectra acquired for the blank filter were used for our background subtraction routine. For each blank filter, the ratio of

the intensity of the fixed element channel window to the backscattered excitation channel window was calculated for each element to be determined for the specific fluorescer utilized. For each sample spectrum acquired, the spectrum background for each element was calculated, using the intensity of the backscattered radiation peak in conjunction with above ratio values established for the blank filter.

d. Next, for each element, the spectral background due to peak overlaps for x-ray lines from nearby elements was calculated. This was accomplished using an iteration process. Individual element x-ray line overlap constants (stored in our XRF program) and initial element x-ray line intensity values (total counts in element channel windows minus the spectral background calculated) were used in the iteration process. The individual element x-ray line overlap constants used were previously established from thin deposits that yielded x-ray lines from only a single element.

e. The total background for each element x-ray line is the sum of the spectral background due to the scattered excitation radiation, SB, and the background due to peak overlaps, PO. The net element x-ray line counts, I, is the total counts, T, in the element channel window minus the total background, SB + PO, as shown in equation 1.

$$I = T - (SB + PO) \quad (1)$$

f. The initial element concentration values (on the ng/cm² basis) are calculated as shown in equation 2.

$$C_i \text{ (ng/cm}^2\text{)} = (I \times C_s) / (I_s \times k) \quad (2)$$

Where,

C_i = initial concentration value, C_s = concentration of the standard (ng/cm²), I = net element x-ray line counts,

I_s = intensity of the x-ray line counts for the standard element on the standard filter, and k = the relative efficiency factor for the element of interest, relative to that for the standard element.

Procedures employed to determine relative excitation/detection efficiency factors are described in Appendix 1.

g. The values of the terms T, SB, PO, I, and C for each element were stored on a floppy disk. Also included for each element was the standard deviation in the counting error reflected in the determination of the value of C. Additionally, the information utilized from the RECDATA file was also written on the same disk with the element information. The date and time the x-ray spectra were acquired were also stored on the disk. All of these values stored on disk were printed on a hard copy which was examined to see that all of the results are within reasonable ranges. Particular emphasis was placed on examination of the intensities of the backscattered excitation radiation peaks. This was to ensure that there was not a malfunction in the total x-ray beam dumps utilized

APPENDIX 1. CALIBRATION OF THE X-RAY SPECTROMETER AND EXAMPLES OF
HARDCOPY OUTPUTS GENERATED XRF PROGRAMS

LIST OF FIGURES

FIGURE

1.1 Plot (Generated by the ELPLOT Program) of the Ratio of Determined to Assigned True Concentration Values for Five Elements, S, K Ca, Ti, and Mn on the Control Filter.

1.2 Plot (Generated by the ELPLOT Program) of the Ratio of Determined to Assigned True Concentration Values For Five Elements, Fe, Ni, Cu, Zn, and Rb on the Control Filter.

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TABLE

1.1 Solvents and Elements or Compounds Used to Prepare Standard Solutions.

1.2 Mean Values Calculated, Using the MEANVAL Program, for the 19 Elements on the Control Filter.

APPENDIX 1

1. Calibration of the X-Ray Spectrometer

a. Absolute calibration of the spectrometer for a single element, Cu, was achieved by using several evaporated gravimetric thin film standards prepared at LBL. Calibration for most of the other elements was achieved using multielement standard solution deposits collected from the fine stage of a dichotomous sampler. The multielement solutions were prepared for aliquots of individual element standard solutions, most of which were prepared by dissolving weighed amounts of high-purity elements in acidic solutions and making to volume. In some cases, dried high-purity compounds were used when the pure elements were not appropriate for weighing. Table 1.1 lists the individual elements or compounds used to prepare standard solutions and the solvent employed.

b. The actual amount of multielement standard solutions collected on individual filters was unknown. However, in most cases, Cu served as the internal standard in the multielement standard solutions, used to determine the relative response of the spectrometer for various elements. The combination of various elements into multielement standard solutions was dictated by compatibility (no precipitation or volatility for a given element) and by minimization of overlapping x-ray lines. Deposit loadings were made thin enough so that x-ray absorption effects were not a consideration. Typical individual element mass loadings were in the 1 to 10- $\mu\text{g}/\text{cm}^2$ range.

c. Standard solutions were not prepared for Ga, Ge, or Se. To achieve these ends, the relative excitation/detection efficiencies determined with the Mo fluorescer, Ti ($Z = 22$) to Sr ($Z = 38$) were plotted versus the respective K x-ray absorption edges of the individual elements. Calibration for the elements Ga, Ge, and Se were made by interpolation from the above curve.

d. For lower atomic number elements, such as S, Cl, K, and Ca, very dilute standard solutions containing a single element with Cu were used. In these cases, the concentration ratios of the lower atomic element to that of Cu were typically in the range of 5:1 to 20:1. This was done to minimize the possibility of x-ray absorption due to particle size effects. Calibration

for Al and Si was achieved using evaporated gravimetric thin film standards. For these latter standards corrections for matrix absorption effects had to be calculated.

e. Estimated total calibration errors for the elements of atomic number 16 (S) and higher were in the range of 1 to 3 percent.

f. Our calibration procedures for most of the elements have been substantiated through the analyses of National Bureau of Standards Reference Standard Materials (SRMS), United States Geological Reference Materials, and Reference Materials from the National Institute of Environmental Studies of Japan. For all of these materials, thin specimens were prepared, and corrections for matrix effects had to be ascertained. Results for many of these determinations have been published. Additionally, we have participated in a number of round robin studies, and our results reported have always been found to be among the best.

g. Estimated total potential calibration errors for Al and Si are 20 percent and 10 percent respectively. The estimated errors for these two elements are large, principally due to the assumptions made regarding the computation of the absorption corrections for the evaporated gravimetric thin film standards.

2. Examples of Hardcopy Outputs Generated for the XRF Programs

Table 1.2 lists the mean values calculated by the MEANVAL program for the 19 elements on the control filter. These determinations were carried out over a nine month interval.

Figures 1.1 to 1.2 are plots of this same set of data. These plots were generated by the ELPLOT program. The ratio of the determined to the assigned true concentration values are plotted versus the number of the control filter run.

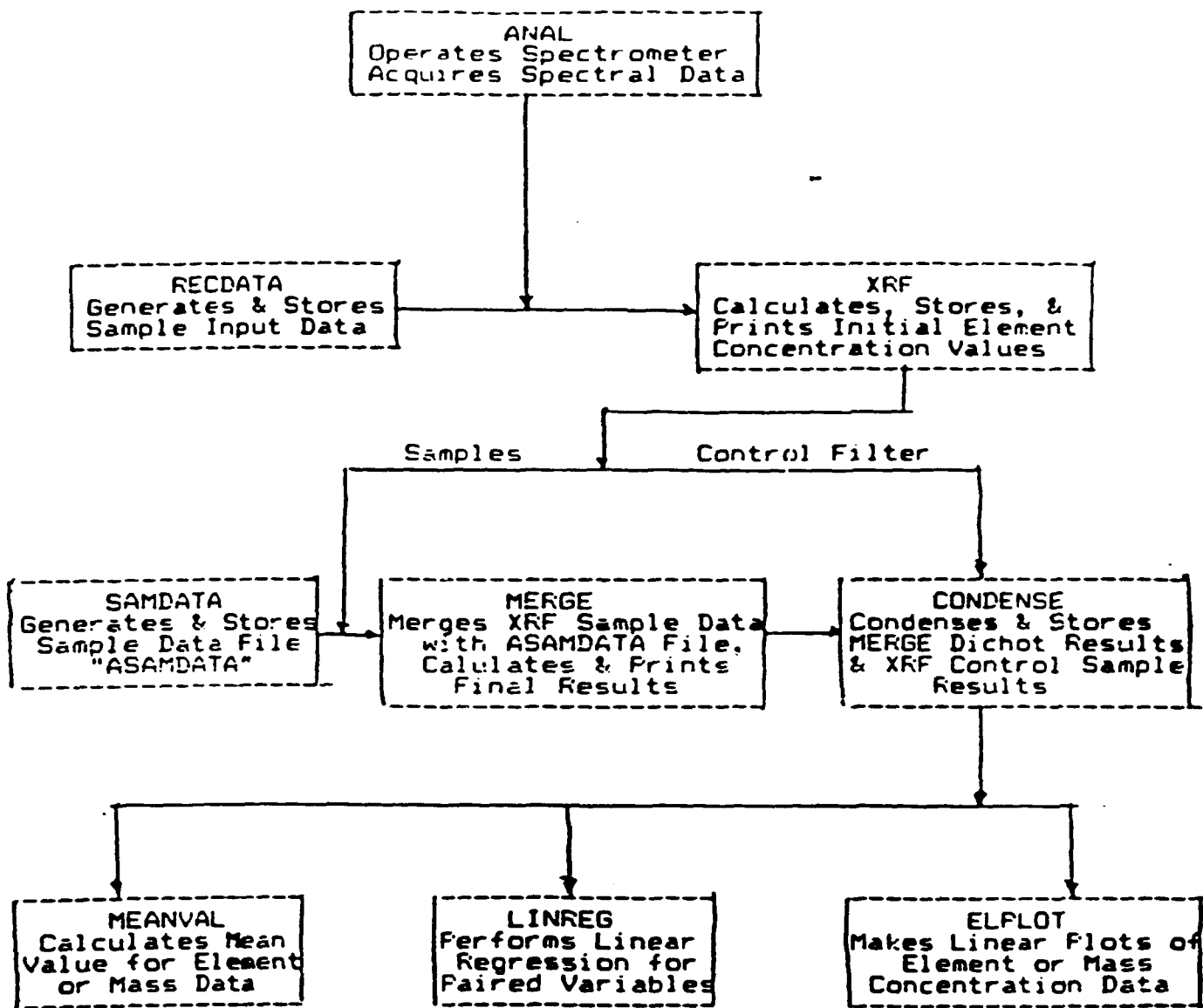


Figure 1. XRF Programs Flowchart

Table 1.1. Solvents and Elements or Compounds Used to Prepare Standard Solutions.

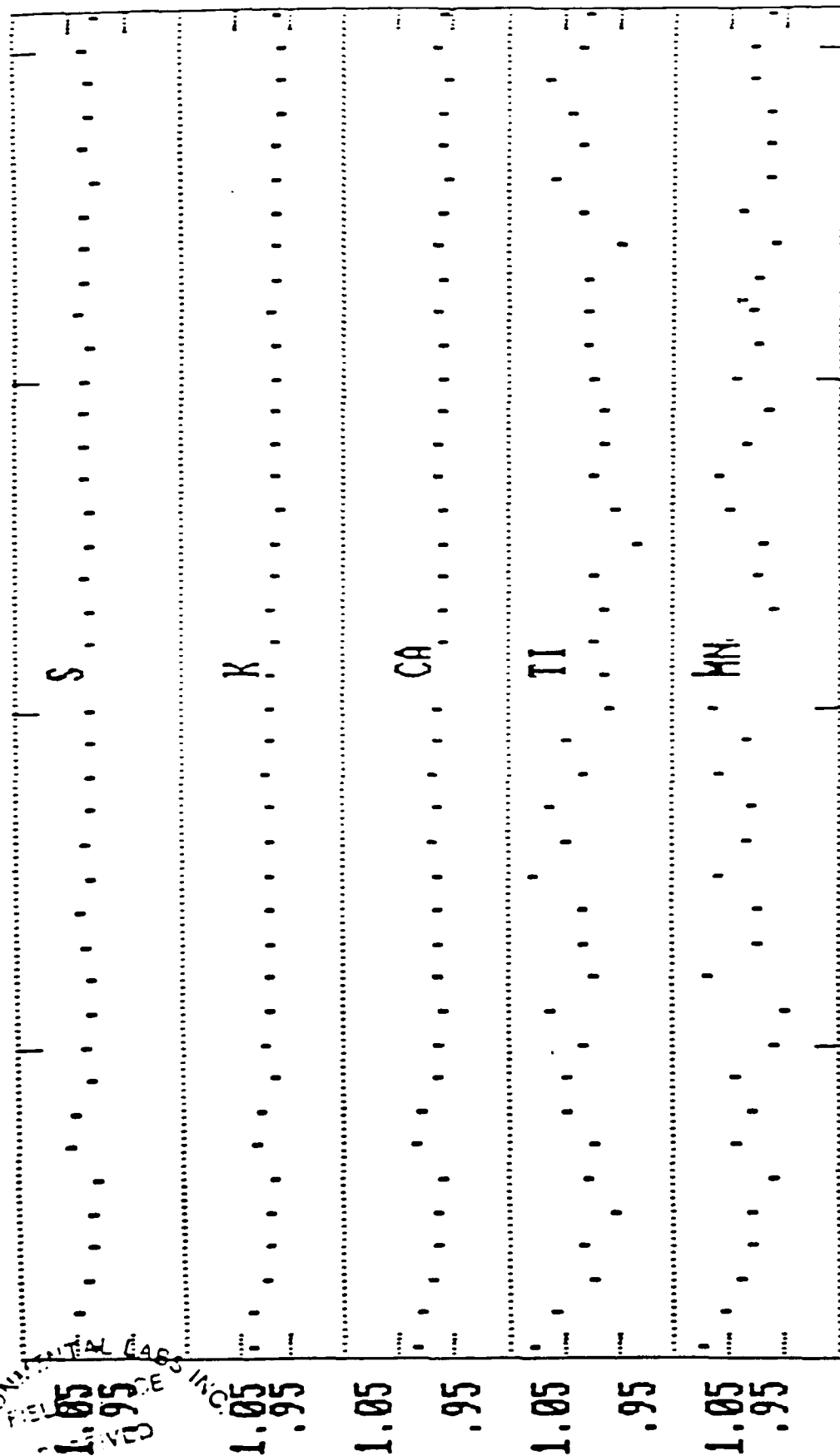
<u>Element</u>	<u>Compound</u>	<u>Solvent</u>
S	$(\text{NH}_4)_2\text{SO}_4$	H_2O
Cl	NH_4Cl	H_2O
K	K_2CO_3	H_2O
Ca	CaCO_3	dil. HNO_3
Ti	Ti	$\text{HF} + \text{HNO}_3$
V	V	$\text{HF} + \text{HNO}_3$
Cr	$\text{K}_2\text{Cr}_2\text{O}_7$	H_2O
Mn	Mn	dil. HNO_3
Fe	Fe	dil. $\text{HCl} + \text{HNO}_3$
Ni	Ni	dil. HNO_3
Cu	Cu	dil. HNO_3
Zn	Zn	dil. HNO_3
As	As_2O_3	dil. NH_4OH
Br	KBrO_3	H_2O
Rb	Rb	H_2O
Sr	SrCO_3	dil. HNO_3
Zr	Zr	$\text{HF} + \text{HNO}_3$
Mo	Mo	HNO_3
Ag	AgNO_3	H_2O
Cd	Cd	dil. HNO_3
Sn	Sn	$\text{HCl} + \text{H}_2\text{O}_2$
Sb	Sb	$\text{HF} + \text{HNO}_3$
I	KIO_3	H_2O
Ba	BaCO_3	dil. HNO_3
Pb	Pb	dil. HNO_3

Table 1.2. Mean Values Calculated Using the MEANVAL Program. for the 19 Elements on the Control Filter.

Group 1 Element	Start File 1 Concentration (ng/cm ²)	ID Cont-5F Error (ng/cm ²)	End File 41 Mean Value for Group (ng/cm ²)	ID Cont-5F Standard Deviation (ng/cm ²)	Number Values
S	15300	600	15466	106	41
K	4440	180	4373	42	41
Ca	4080	160	3996	38	41
Ti	1030	50	1036	30	41
Mn	409	18	412	11	41
Fe	2870	120	2850	29	41
Ni	251	11	252	5	41
Cu	57	7	53	4	41
Zn	1380	60	1375	10	41
Rb	220	9	219	4	41
Sr	373	15	371	4	41
Pb	2150	90	2165	20	41
Zr	93	7	89	5	41
Mo	454	19	448	9	41
Ag	97	6	93	4	41
Cd	369	16	366	7	41
Sn	457	20	455	11	41
Sb	231	11	229	6	41
Ba	502	30	510	28	41

Determining Values / Concentration

ENVIRONMENTAL
RESEARCH
LABORATORIES
INC.
10000
10000



10 20 30 40

OK

Control File No.

Figure 1.1

Plot (Generated by the ELPLLOT Program) of the Ratio of Determined to Assigned True Concentration Values For Five Elements, S, K, Ca, Ti, and Mn on the Control Filter

Determined values / Concentration

ENVIRONMENTAL
FIELD OFFICE
RECEIVED
JAN 26 1983

FE

1.05
.95

NI

1.15

CU

.85

ZN

1.05

.95

RB

1.05

.95

10

20

30

40

Control File No.

OK

Figure 1-2

Plot (Generated by the ELPIOT Program) of the Ratio of Determined to Assigned True Concentration Values
For Five Elements, Fe, Ni, Cu, Zn, and Rb on the Control Filter

Literature Cited

- 1.1 F.S. Goulding, J.M. Jaklevic, and B.W. Loo, "Development of Air Particulate Monitoring Systems", Lawrence Berkeley Laboratory, Berkeley, CA UCID-8090 (1978)

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APPENDIX C. QUALITY ASSURANCE PROJECT PLAN

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QA PROJECT PLAN FOR
OPEN BURNING AND OPEN DETONATION
THERMAL TREATMENT EMISSIONS STUDY
BANGBOX TEST SERIES

APPROVAL: Program Manager ORIGINAL SIGNED Date _____
MacDonald B. Johnson

QA Official ORIGINAL SIGNED Date _____
Gary M. Booth

Project Officer ORIGINAL SIGNED Date _____
Kenneth Jones

Technical Coordinator ORIGINAL SIGNED Date _____
H. Smith Broadbent

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SECTION 1. Program Description

1.1. Current estimates of obsolete and/or unsafe explosive materials approach 200,000 short tons. The most appropriate way to dispose of most of these materials is by open burning (OB) and open detonation (OD). Because there is limited empirical data on the generation of particulates and criteria pollutants from OB/OD operations, a well-defined research program is critically needed.

1.2. Accordingly, the purpose of this preliminary study is to verify and validate OB/OD test technology. This technology will include instruments, sampling equipment and procedures to be used aboard a fixed-wing aircraft (FWAC) on larger scale outdoor trials as well as sampling analysis methods and QA/QC procedures. These preliminary tests, referred to as the BangBox tests, will be conducted at Sandia National Laboratories (SNL) in Albuquerque, New Mexico.

1.3. This program brings together the expertise of scientists from Alpine West Laboratories (AWL), Battelle-Columbus Division (BCD), SNL, Oregon Graduate Center (OGC), Sunset Laboratories (Sunset), and Lawrence Berkeley Laboratories (LBL). All laboratories are preparing Letters of Instruction (LOI). Instructions for preparing LOI were sent out to all the laboratories during the second week of November 1988 (Appendix I contains a copy of Instructions for Preparing LOI) by Environmental Labs Inc. (ELI). Many of these data collection and analyses required methods development and all analytical methods did not fall under U.S. E.P.A. (Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Monitoring and Evaluation Branch) Preferred Methods.

1.4. Complete copies of the test design plan may be obtained from Andrulis Research Corporation, Salt Lake City, Utah.

SECTION 2. Project Organization and Responsibilities

All personnel will come from DOD, AWL, Andrulis Research Corporation, BCD, SNL, OGC, Sunset, and LBL. Other scientific consultants may be called on periodically as the project progresses. Quality assurance will be done by personnel from ELI and EPA.

SECTION 3. QA Objectives in Terms of Precision, Accuracy, and Completeness

3.1. There will be several detonations of 0.5 pounds of TNT each under a neoprene bubble (the BangBox) sometime during the last week of November and during the 2nd week of February 1989. All of the samples to be analyzed will be collected on-site. SNL will be responsible for coordinating the detonations. Dugway Proving Ground (DPG) will be responsible for obtaining soil samples (for spiking experiments) to be analyzed by AWL and BCD.

3.2. The following QA objectives for completeness, accuracy, and precision will be used in the design of this study:

3.2.1. Completeness. Seventy-five percent of all possible measurement data should be valid.

3.2.2. Accuracy. All gas and organic exotic analysis results should agree within ± 25 percent of known spiked concentrations. However, these limits may have to be modified if certain analytes cannot be measured at these limits. Spiking of the analytes into the canisters, resins, and DPG soil

samples will be conducted by EPA; or the spiking of the soil and resin samples can be conducted by the technical coordinator or other scientists agreed upon by ELI QA and EPA.

3.2.3. Precision. Since many of these procedures require methods development, good estimates of precision will be determined by the individual laboratories and subsequently reviewed by QA officers and EPA for acceptance and verification. These estimates of precision may include analysis of standards, surrogates, replicates, and the samples themselves.

SECTION 4. Sampling and Analysis Procedures.

4.1. Sampling and analysis involves basically three parts. These are: (1) sampling and analysis of soil samples from DPG which are subsequently spiked with known concentrations of analytes, (2) washed resins which are spiked with known concentrations of analytes, and (3) actual sampling and analysis of particulates and targeted emissions from within the neoprene bubble at SNL following each detonation. The soil sampling and analysis will be according to instructions in LOI prepared by AWL.

4.2. A QA officer and representatives from DPG and AWL will sample pristine soil samples from Dugway prior to the BangBox test. A collection report will be filled out for each soil sample. The samples will be placed immediately in dry ice and returned to AWL for drying, homogenizing, and weighing. A total of 15 of these 250-g soil samples will be tracked by utilizing a shipping list and then sent to the EPA for spiking. Each set of five samples will contain one control, two dosed at one concentration and two dosed at another concentration. The exact concentrations, unknown to the analytical laboratories involved, will be determined in consultation with scientists on the project.

One set of five samples will be sent to AWL, one set to BCD, and one set will be retained by the EPA. Appropriate chain of custody papers will be provided to EPA QA personnel for each sample. ELI personnel will assist in shipping these soil samples to the EPA and insure that the integrity of the samples is maintained at the point of shipment.

4.3. Two types of resins, XAD-2™ and PoraPak R™, will also be spiked by EPA representatives for determinations of concentration of recovered compounds. The choices of analytes and concentrations will again be determined by consultation with the appropriate scientists. A total of nine XAD-2™ samples and nine PoraPak R™ samples will be sent by AWL to EPA utilizing a Collection Report and Shipping List. A given set of three samples of XAD-2™ resins will have one control, and one solution (at a given concentration) spiked in duplicate. This procedure will be repeated for the PoraPak R™ samples. Sets of three samples for each resin will be sent to AWL and to BCD, and one set will be retained by the EPA. Thus, each laboratory will receive five soil samples and six resin samples. The known concentrations of spiked soil and resin samples will be provided to the Program Manager.

4.4. AWL will utilize methods of supercritical fluid chromatography/mass spectrometry (SFC/MS) to determine the analyte concentration while BCD will use gas chromatography/mass spectrometry (GC/MS) methods. A formal report by the EPA on the comparison of the two methods will be provided to the Program Manager and to ELI.

4.5. In addition to the soil and resin samples, three 6-L canisters will be spiked by EPA with appropriate volatile organics (one control and one spiked in duplicate) and subsequently analyzed by OGC. Two 32-L tanks will be identically spiked by EPA with exotic organics and sent to OGC

for reflux extraction. This extract will be sent to AWL which will split the unconcentrated sample into thirds. Then one-third will be sent to BCD, one-third will be retained by AWL for analysis, and one-third will be sent to EPA for archiving. Upon completion of the analyses by the laboratories, a formal report will be sent to the EPA and a copy to the Program Manager. EPA will then send a report to the Program Manager which compares the laboratories' results with the concentration values of the original spiked samples.

4.6. At the appropriate time, EPA will also supply a sample of the spiking solution to both laboratories (BCD, AWL).

4.7. In addition to the spiked samples and certain comparisons discussed above, other samples such as real-time samples, OGC gas samples, and AWL samples will be monitored. Also, assessment of real-time instrument performance at SNL will be additionally completed by a team of EPA QA auditors. Instrument performance, QC procedures, and general analysis techniques will be monitored during the audit visits at each laboratory.

4.8. Tables 1.3 through 1.6 of the test design plan summarize the analysis, level of effort, assay methods, and responsible laboratory for the experiments to be completed during the preliminary test at SNL. Table 1.7 of the test design plan summarizes the contaminant/combustion compound list for the study.

SECTION 5. Sample Custody

5.1. Sample custody forms are required for all soil samples and resin samples for the spiking experiments. Sample custody forms will also be completed for the receipt, storage, and use of the TNT samples received by SNL. Prior to departure to the individual laboratories, sample custody forms will be completed and given to a QA officer) on all BangBox samples. In-house tracking forms (History of an Official Sample) will be used by each laboratory. QA/QC aspects of procedures used in handling the actual samples themselves will be assessed by comparing LOI procedures to operations.

SECTION 6. Calibration and Logbook Procedures.

6.1. All continuous gas monitors will be calibrated according to the manufacturers' recommended procedures. Calibration curves for selected organic exotics and inorganics will also be prepared by the laboratories. Quality criteria for the calibration curves shall be outlined by each laboratory and recorded. Correlation coefficients and/or tables of residuals may accompany each set of data. The frequency of calibrations should also be noted as part of the LOI. In addition, technical instruments such as balances and chromatographs should be calibrated periodically within specified time periods and documented. The quality criteria for calibration and maintenance of the equipment will be maintained by the individual laboratory's QC program. Daily logbooks will be kept on all instrumentation.

SECTION 7. Data Management and Archiving of Raw Data.

7.1. All of the data collected during the QC experiments and the BangBox investigation from each laboratory, including all calibration data and logbook data, will be sent directly to the Data Management Center (DMC) at ELI. This includes all appropriate summary data to be used in the final report. The DMC will be responsible for copying these data and sending the originals to a final QA storage area. Each data page will be stamped by the QA DMC and the QA records

custodian for final storage. When the entire study by a given laboratory is completed, sent to ELI, and stamped, copies of these data will be turned over to the DPG project officer for preparation of the final report. The originals will be placed in the QAU. Complete copies of the data will be eventually stored in the archiving facility at the Smith Family Living Center at Brigham Young University (BYU), or other appropriate facilities agreed upon by the Program Manager. Where possible all data should be sent to ELI on an IBM compatible disk. Some raw data, such as that reported from SNL, will be sent to ELI on a disk that is compatible with HP-300 computers. Magnetic tapes are also acceptable for storage of raw data; however, back-up discs of all raw data in addition to the magnetic tapes are strongly encouraged.

SECTION 8. Data Analysis, Validation, and Reporting.

8.1. Data analysis will be the responsibility of Andrulis Research Corporation. As indicated in Section 7.0, the data received from each laboratory will be received by ELI DMC and then sent to Andrulis. Upon receipt of the data, sample custody forms will be filled out and signed by both the QA and Andrulis personnel. Once the final report is completed, a QA team (composed possibly of EPA and ELI QA officers) will meet with Andrulis and spot-check the raw data against that which is found in the final report. Upon satisfactory completion of the QA audit, a formal QA statement will be provided and placed in the final report.

SECTION 9. Internal Quality Control Checks and Frequency

9.1. Each laboratory will be expected to have an internal QC program. These QC checks will be written up in the form of LOI. Basically, these are a listing of the operational checks, the control limits for initiating corrective action, and the planned corrective action. Examples of items that might be considered include:

9.1.1. Replicates

9.1.2. Spiked samples

9.1.3. Split samples

9.1.4. Control charts

9.1.5. Blanks

9.1.6. Internal standards

9.1.7. Quality control samples

9.1.8. Calibration standards and devices

9.1.9. Reagent checks

SECTION 10. Performance and System Audits

10.1. Personnel from ELI will visit each laboratory no more than once while analyses are being performed to monitor and assess the capability and performance of all instrumental and analysis systems, and assess the adherence to approved procedures. Each laboratory director will be contacted prior to each visit. A Quality Assurance Unit Inspection Form (OB/OD Site Visit Worksheet) will be used by each QA inspector. These completed forms shall be used to write a formal letter to the Principal Investigators and a copy of these letters will be sent to the OB/OD Program Manager. Copies of all audit reviews will be retained in the DMC and the QA storage file. The results of the scheduled spiking of the analytes by the EPA in the soil, resins, 6-L canisters and 32-L tanks may also be part of the audit.

10.2. In addition, an EPA audit team will provide a performance audit on the real-time instruments at SNL. The measurements will include the parameters O₃, SO₂, NO_x, and others as time permits.

SECTION 11. Preventative Maintenance.

Preventative maintenance tasks and schedules recommended by the manufacturers of the gas analyzers, chromatographs, mass spectrometers, and other technical equipment will be followed. Spare parts such as detector heads, septa, columns, and cylinder gases should be readily accessible during the project for daily checks and recalibrations. These procedures also should be defined in the LOI.

SECTION 12. Specific Procedures For Routine Assessment of Data Precision, and Accuracy

12.1. There are routine procedures used to assess the precision and accuracy of the measurement data. If appropriate, these procedures should include the equations to calculate precision and accuracy, and the methods used to gather data for the precision and accuracy calculations.

12.2. Examples of statistical procedures to be selected by Andrulis Research Corporation are central tendency and dispersion, measures of variability, significance tests, Confidence limits, and testing for outliers.

SECTION 13. Quality Assurance Reports to Management.

13.1. A report, covering the activity for each laboratory, will be provided to management as described in section 10. These reports may include:

13.1.1. Assessment of measurement data accuracy, precision, and completeness.

13.1.2. Results of performance audits

13.1.3. Results of system audits

13.1.4. Significant QA/QC problems and recommended solutions

13.2. The ELI QA Unit will be responsible for preparing these reports. And, as already mentioned, a separate QA section will be provided in the final report which summarizes data quality information contained in the reports.

SECTION 14. Summary

The goal of this QA project plan is to provide the policies, objectives, functional activities, and specific QA/QC activities associated with the LOI and which are designed to achieve data quality. The heart of the QC aspect of the QA project plan is for each laboratory to submit LOIs to the QA unit in order to establish written, documented procedures. LOI were chosen instead of Standing Operating Procedures (SOP) for this project because much of the SNL preliminary phase data collection will be methods development. The flow of information will come from each parent laboratory to the Data Management Center to a QA storage file to DPG to Andrulis Research Corporation (who prepares the report), back to the QA Unit and finally to the Program Manager. During the 2-4 month effort, there will be a site visit to each laboratory to insure QA/QC compliance.

SECTION 15. Glossary of Terms

- Audit** A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which quantitative data are independently obtained for comparison with routinely obtained data in a measurement system, or (2) system audits of a qualitative nature that consist of an on-site review of a laboratory's QA/QC system and physical facilities for sampling, calibration, and measurement.
- Data Quality** The totality of features and characteristics of data that bear on their ability to satisfy a given purpose. The characteristics of major importance are mainly accuracy, precision, and completeness. These characteristics are defined as follows:
- Accuracy** The degree of agreement of a measurement, X , with an accepted reference or true value, T , usually expressed as the difference between the two values, $X-T$, or the difference as a percentage of the reference or true value, $100 (X-T)/T$, and sometimes expressed as a ratio, X/T .
- Precision** A measure of mutual agreement among individual measurements of the same property, usually under prescribed, similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist, depending upon the "prescribed similar conditions."

Completeness A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions.

Data Validation A systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, certification, and review.

Performance Audits Procedures used to determine quantitatively the accuracy of the total measurement system or component parts thereof.

Quality Assurance (QA) The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Assurance Project Plan An orderly assembly of detailed and specific procedures which delineates how data of known and accepted quality are produced for a specific project.

- Quality Control (QC) The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. Often, QC procedures are referred to as those standards of performance that are checked within a laboratory.
- Letter of Instruction (LOI) Written documents which generally outline an operation, analysis or action whose mechanisms are generally prescribed and which are commonly accepted as the methods for performing certain routine or repetitive tasks. The LOI are more general than the Standing Operating Procedure (SOP).
- Chain of Custody A procedure for preserving the integrity of a sample or of data (e.g. a written record listing the location of the sample/data at all times).

BangBox QAPP
Revision - 2
2 November 1988

Appendix I

Procedures for Writing Letters of Instruction (LOI)

Procedures for Writing Letters of Instruction (LOI)

SECTION 1. Purpose

QA/QC practices require that all routine activities having an impact on data quality be fully documented. These day-to-day routine practices should be itemized in a clear, explicit, somewhat detailed, step-by-step manner so that most individuals could follow them in a uniform and consistent manner.

SECTION 2. Scope

2.1. LOI should be prepared for all routine activities associated with the OB/OD project. Such activities may include:

2.1.1. Field or Laboratory Sampling

2.1.2. Field or Laboratory Analysis

2.1.3. Instrument or Method Calibrations

2.1.4. Preventative and Corrective Maintenance

2.1.5. Internal QC Procedures

2.1.6. Site Selection

2.1.7. Sample Preparation and Storage

2.1.8. Preparation and use of Spiked Samples

2.1.9. Instrument/Equipment Selection and use

2.1.10. Determination of Detection Limits and Limits of Quantification

2.1.11. Sample Handling and Transportation

2.1.12. Conduct Performance Audits

2.1.13. Data Reduction and Analysis

2.2. For the "Bang-Box" portion of the OB/OD project, LOI can be more general than SOP.

2.3. Once the LOI are written, they will be reviewed for adequacy by the QA Officer and Technical Coordinator, and kept in each Laboratory's files and made available during QA visits.

LOI may also be changed or revised during the study to conform with the actual work effort. However, these changes should be clearly documented and the old LOI replaced with the new one (see document control section below).

SECTION 3. Format

3.1. The format and outline of LOI may vary somewhat depending on the activity. However, all LOI should contain the following:

3.1.1. Title

3.1.2. Outline of procedures

3.1.3. References (if appropriate)

3.1.4. Document control block in upper right hand corner

3.1.5. Signature line for project officer and signature line for QA

SECTION 4. Document Control

Each page of the LOI should contain a document control block in the upper right corner containing: LOI number, date of issue, revision number, and page number similar to that used for this document.

SECTION 5. Where to Send LOI

5.1. A copy of each LOI should be kept in the originating laboratory and a signed copy should be sent to:

Dr. Gary M. Booth
Environmental Labs, Inc.
1125 South 550 East
Springville, Utah 84663

5.2. All LOI should be received by Nov 28, 1988. If you have any questions, you may call Gary Booth at (801) 378-2458.

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APPENDIX D. QUALITY ASSURANCE AUDIT REPORTS

Quality assurance activities were conducted throughout the execution of the BangBox Test. Visits were made to test sites during detonation and burning activities, and to contracted laboratories which assayed samples obtained during the testing. Results of these visits are contained in the 13 enclosed reports.

ENCLOSURE	TITLE
1.	Memorandum, Floyd W. McMullin, 17 December 1988, subject: Report on site visit to Sandia National Lab [sic].
2.	Memorandum, 28 December 1988, Todd Parrish, subject: Battelle-Columbus Site Visit.
3.	Memorandum, 24 January 1989, Floyd McMullin, subject: Report of Site visit to Sunset Laboratories 23 Jan 89 [sic].
4.	Memorandum, 24 January 1989, Floyd McMullin, subject: Report of Site visit to Oregon Graduate Center [sic].
5.	Memorandum, 24 January 1989, subject: Miscellaneous items from OGC site visit [sic].
6.	Memorandum, 25 January 1989, Todd Parrish, subject: AVL Site Visit.
7.	Memorandum, 31 January 1989, Floyd W. McMullin, subject: Report of site visit to Lawrence Berkley Lab (LBL) [sic].
8.	Memorandum, 08 February 1989, Floyd McMullin, subject: Addendum to site report for Oregon Graduate Center [sic].
9.	Letter MD-77B, February 15, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed results of OB/OD audit at Sandia National Laboratories.
10.	Letter MD-77B, March 2, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed report of spiked samples.
11.	Letter MD-77B, March 7, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed report of CO and CO ₂ audits.

12. Memorandum, 12 May 1989, Dr. Gary Booth, subject: Interim Report on Site Visits.
13. Memorandum, 12 May 1989, Floyd McMullin, subject: Sunset Laboratory site visit 9 May 1989 [sic].

MEMORANDUM, FLOYD W. MCMULLIN, 17 DECEMBER 1988, SUBJECT: REPORT
ON SITE VISIT TO SANDIA NATIONAL LAB [sic]

ENCLOSURE 1

MEMORANDUM

TO: Environmental Labs, Inc.
ATTN: Dr. Gary Booth
FROM: Floyd W. McMullin *Mac*
RE: Report of site visit to Sandia National Lab (SNL)
DATE: 17 December 1988

The following is my report of the site visit to SNL on the 16th of December 1988. I met with Wayne Einfeld and spent several hours discussing various aspects of the project and checking the assorted areas of concern for quality assurance. For purposes of review and discussion I will basically duplicate the content of my logbook. The only changes that will be incorporated would be spelling and grammatical corrections, and if appropriate, more definitive explanations for clarity if I feel it is warranted.

The areas that I've written about basically correspond to the listing of components of a good quality assurance plan as listed in the EPA guidelines, and in the directions for preparation of a QA plan (Appendix to our QA plan).

LOI STATUS: All pertinent information on real-time instrumentation was well covered in the initial LOI submitted during the initial phase of the test. Those analyses which are done after the fact (HCN, NH_3) will be referenced to standard analysis procedures. This will be done in addition to be appended to the existing LOI. Wayne has not completed these items at this time due to time limitations and the need to finish the real-time data analyses from the first test prior to meeting planned for Salt Lake City on 4 January 89. This is essential as it is planned to review data from the first test to determine whether or not changes in methodology need to be incorporated prior to the second test series scheduled to commence on 9 January 1989.

FIELD/LAB SAMPLING: All samples collected are assigned an in-house standardized number (These are referenced to the ELI numbers on the collection reports and shipping lists). The general flow of the filter samples is; (1) Filter is weighed. (2) Filter is placed in a filter holder. (3) Holder with filter is placed in travel case for transport to the test site. (4) As filters are used during the test, times and filter numbers are logged. (5) Filters are returned to the lab after the test, and removed from the filter holder. (6) As each filter is removed from the holder it is again weighed (data on pre and post test weights, and filter numbers are logged in a computer system in the lab for storage and retrieval). (7) Filters

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DEC 28 1988

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are then transferred to 47mm petri slides for storage. (8) After being placed in the petri slides all filters are stored together in a filing cabinet in the lab area.

In addition to the actual sample filters there is also a blank filter which also goes through all of the above steps, with the exception of not having been used for sampling.

The lab area (as well as SNL itself) is a limited access area through the use of a cipher type door lock. The storage at this facility is only temporary as all filters are forwarded to other labs for actual analysis. Consequently no sign-in, sign-out system is utilized as they are placed in storage, and then removed for shipment. At the time of shipment they are accompanied by individual collection reports and a shipping list.

Wayne asked me about the need to adding the ELI sample number in addition to the standard numbering system on each sample. Based on the small size of the individual containers, and the possibility of creating confusion, and the fact that the standard numbers are referenced to the ELI numbers (on the collection reports and the shipping lists). I told him I did not feel this would be necessary.

Due to the type of samples involved no special preservation methods are needed for the filters. They are stored at ambient room temperature.

Handling of the NH_3 and HCN samples is somewhat different since these materials are of a type which may deteriorate over time. Their handling is as follows: (1) Upon removal from the bubbler they are sealed on site. (2) They are transported to the SNL Analytical Chemistry lab for analysis. (4) Upon arrival in the lab at a special sample receiving area, in-house sample identification and custody paperwork is initiated (The system utilized appears to be a very good system). (5) Upon completion of this process they are transported (next room over in the same building) to the analytical lab for actual analysis.

There are several factors relating to these samples which are important to note. First, due to the time critical nature of these samples, they are analyzed shortly after receipt in the lab. Total elapsed time from sampling to analysis <1 day, and would normally be accomplished on the same working day as when the sample was obtained. Secondly, as the entire sample is consumed during the analysis process there is no need for sample storage, custody, etc. concerns after the analysis.

FIELD/LAB ANALYSIS: All real time raw data is stored electronically during the actual test for later analyzation utilizing computer. This electronic record also records time data to allow for verification for sampling times in the event they should be missed in the research journals. Wayne stated this became even more exact when working in the Otter as the system recorded when switches are thrown and other pertinent information.

As noted earlier the lab analyses performed in the analytical laboratory are utilizing standardized procedures which will be referenced in the completed LOI.

INSTRUMENT/METHOD CALIBRATION: All are as per the LOI and equipment manufacturers requirements/recommendations.

PREVENTIVE/CORRECTIVE MAINTENANCE: As per equipment manufacturers requirements.

INTERNAL QC PROCEDURES: Wayne is serving as the internal QC for this project. Although SNL has an in-house QA unit, he feels it would be next to impossible to bring this unit in due to the extremely rapid timetable we are attempting to complete this work in. I would tend to agree with that assessment.

As noted, blanks of all filters have been made. Wayne also plans on contacting the EPA to see if they can supply the thin film standard to utilize as a spiked sample for LBL. LBL would not be made aware of the constituents of the sample. Wayne is also planning on providing Sunset Labs with spiked filters. Prior to doing so he is planning on meeting with Robert Cary from that lab to determine the appropriate compound to use. Sunset lab would not be made aware of the amount of material the filter would be spiked with.

SAMPLE PREPARATION AND STORAGE: As previously noted no after analysis storage is needed for the NH_3 and HCN as the sample is consumed in the analysis process.

PREPARATION AND USE OF SPIKED SAMPLES: As noted in the internal QC procedures section.

INSTRUMENT/EQUIPMENT SELECTION AND USE: Selection of the instruments and equipment was determined prior to testing, by the technical committee and the scientists involved, and is not an appropriate item for review for myself. So far as I have been able to determine it would appear they are being utilized according to accepted practice and/or as specified in the LOI.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION: This information accompanies individual laboratory analysis reports.

SAMPLE HANDLING AND TRANSPORTATION: Filter samples are stored in lab (limited access) prior to shipment. Mode of shipment to LBL and Sunset is Federal Express. All other samples are hand carried within SNL to the appropriate facility.

DATA REDUCTION AND ANALYSIS: This will be specified in the completed LOI.

This completes the evaluation based on the QA plan requirements. The material following covers various areas of our interest as specified in their headings.



LOGBOOKS: As previously verified on site during the first phase of the testing, calibrations for the real-time instruments was performed as specified in the LOI and recorded in the equipment log books. Daily entries for zeroing and span checks are recorded in research journals.

I was not able to review the calibration information on the apparatus used in the analytical lab. This was due to the inability to locate the contact person (Charles Gray) for this area. As I did not want to spend an inordinate amount of time on this item I discussed this with Wayne and we decided it could be arranged to have this information available during our next site visit.

PERSONNEL WORKING WITH SAMPLES: The following is a list of all personnel having contact with the samples derived from the tests.

Wayne Einfeld	(505)844-4143	SNL Division 6321
Brian Mokler	(505)299-7610	SNL Division 6321 (Contract)
Dennis Morrison	(505)844-3376	SNL Division 6321
Charles Gray	(505)844-7065	SNL Division 3311 ¹
Lori Maestas	(505)844-7760	SNL Division 1822 ²

- (1) Analytical Chemistry Lab
- (2) Electronic Optics and X-Ray Analysis Division

BUILDING DIAGRAMS: These are considered sensitive material and are not generally released. I personally don't see any pressing need for these. The labs I was in were generally a rectangular room without windows, with equipment on lab benches around the perimeter, and if large enough on additional benches out in the room.

RESEARCH JOURNALS: Those I have seen appeared intact with entries in ink.

LAB TRACKING: Analytical lab utilizes in-house standard forms. The SEM lab also keeps track of samples in their care. Other than that, they're not needed as other samples are shipped out for analysis.

OVERALL ASSESSMENT/RECOMMENDATIONS: I feel that SNL utilizes acceptable procedures overall. The only items left pending were the review of the analytical lab instrumentation logbook/file, and the LOI which is still in the process of completion.

Based on the areas I evaluated today I would have no recommendations for changes at this time. Areas where we were not supplied with information in the initial LOI were discussed with Wayne and it was decided these items would be included in the completed LOI.

Upon reflection about the visit there is one additional area which I feel should be checked at our next visit. That is where all of the electronically stored data is kept, its security, and

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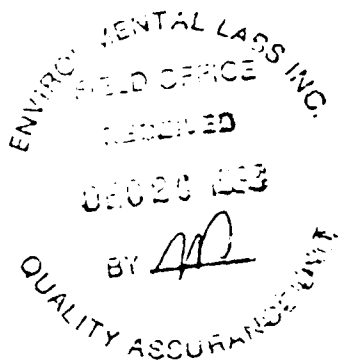
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whether backup files are maintained. This was an oversight on my part.

I would recommend that more than one additional QA site visit to this facility would for the most part be an unnecessary expenditure of funds. I base this on the fact that all on site analytical analyses are completed almost immediately after test completion, or the samples are shipped to other facilities for their analysis. The only ongoing analyses would be the computer analyses of the real-time data from the test, which does not really lend itself to any valid quality audit.

I would recommend that the next site visit be planned for the last part of January or the first part of February 1989.



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DEC 26 1988

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MEMORANDUM, 28 DECEMBER 1988, TODD PARRISH, SUBJECT: BATTELLE-
COLUMBUS SITE VISIT

ENCLOSURE 2

MEMORANDUM

TO: Dr. Gary Booth
FROM: Todd Parrish *[Signature]*
SUBJECT: Battelle-Columbus Site Visit
DATE: 28 December 1988

Following is a report of the 12/28/88 site visit of the Battelle-Columbus Laboratory. I first contacted Larry Slivon on 12/23/88, and set up the 12/28/88 site visit. Upon arrival, Larry Slivon and I discussed various aspects of quality assurance. This report then is a synopsis of those points covered.

The areas of quality assurance that are covered are those that are included in the EPA quality assurance plan. The format is according to the outline organized by Floyd W. McMullin.

LOI STATUS

The LOI are very complete and contain the various procedures that are being performed at BCD. They clearly explain the methods of receipt, storage, sample preparation, and sample analysis. From my observations, the LOI are being followed. The LOI are being revised as some changes need to be implemented. The internal quality control checks are also explained in the LOI.

FIELD/LAB SAMPLING

All samples that are received are given a BCD internal number. That number is easy to determine as it corresponds to a specific notebook, page, and line. This logbook is maintained by Dave Oiler. The logbook also references the ELI number. The samples were received from AWL in sealed glass containers. Upon receipt, the in-house number is assigned, and an aliquot is made from 25% of the sample. All analyses will be done with this portion. The remainder is frozen for future reference or use. All freezers that will hold the samples have a sheet for each individual sample that will track that sample. The sample is signed in and out by an individual, and also provides for a entry of how much was used.

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FIELD/LAB ANALYSIS

The samples will be analyzed by three different means: Negative Ion Procedure (6 target compounds), Positive Ion Procedure (8 target compounds), and an Electron Impact Method. All of the resulting data will be stored on a mainframe computer. It will first be stored on tape and then backed up on disc. At the time of my visit, not much analysis had been accomplished; but they had begun to analyze 5 samples (ELI 0203, 0225, 0205, and 0215). The analysis will be done according to the revised LOI.

INSTRUMENT/METHOD CALIBRATION

The instruments are calibrated as required by the internal quality assurance requirements and the LOI. The individual instruments each have a logbook that contains all work done with the instruments.

PREVENTIVE/CORRECTIVE MAINTENANCE

Corrective maintenance is done when needed. It is accomplished by the individual technicians. Preventive maintenance is done regularly.

INTERNAL QC PROCEDURES

Larry Slivon or his associate Jean Czuzwa will be in charge of the quality control for this project. There is an in-house quality assurance group that will act in an advisory role. The QC advisor is Romona Mayer.

At the beginning of the day a spiked sample will be run. This sample will contain all the target compounds, blanks (internal standard only) will also be run daily. The OB/OD samples will be analyzed using the same instrumental conditions as the standard. The EPA spiked resin and soil samples will also be analyzed. These results will be compared with those of AWL and the EPA.

SAMPLE PREPARATION AND STORAGE

The samples will be prepared as explained in the LOI and will be stored in freezers. Larry Slivon mentioned that the freezers will not be locked because of the amount of people that need access to the samples. They are in locked rooms at night though. The freezers all have daily monitoring sheets that include the temperature. The samples are presently stored in a temporary freezer as the permanent freezer is being thawed out.

PREPARATION OF SPIKED SAMPLES

Each sample will be spiked with an internal standard as indicated by the LOI. The spiking will be done by Dave Oiler.

INSTRUMENT/EQUIPMENT SELECTION AND USE

The equipment used is four years old and in very good condition. Larry Slivon does most of the maintenance. It is not in my expertise to determine the quality or efficiency of the equipment. From what was observed, the instruments were used according to the LOI. The instruments were in a very clean room that was elevated from the floor to eliminate static interference.

DETERMINATION AND DETECTION LIMITS AND QUANTIFICATION LIMITS

Quantification calibration will be performed using a single point standard analyzed at the beginning of each day. These points will become more definite as more analysis is accomplished. The detection limits will be determined from a plot of relative response and concentration of target compounds. This will be determined with more accuracy as the analysis proceeds.

SAMPLE HANDLING AND TRANSPORTATION

The sample custody was explained earlier in the Field/Lab Sampling section of the report. All the samples are carried by hand within the BCD facilities.

DATA REDUCTION

This will be done on the Direct Voss samples before the Salt Lake City meeting in January. All others will be done later and will be included in the report.

This concludes the evaluation required by the EPA Quality Assurance Plan. The following material covers various areas of interest relating to the Quality Assurance as indicated by their heading.

LOGBOOKS

There are two types of logbooks in use: personal project notebooks, and instrument notebooks. If an analysis is done on a sample extract, it will be recorded in both. As far as I can determine from my examinations of the notebooks, they are maintained very well. The writing is in ink, but not always black as specified by the EPA. The daily calibrations are all recorded.

PERSONNEL

The following is a list of the personnel at BCD that will be involved in the OB/OD project.

Larry Slivon	Principal Research	(614) 424-4274
Jean Czuzwa	Associate	(614) 424-7936
Jane Chuang	NCI-SIM	(614) 424-5222
Mark Bower	EI-MS	(614) 424-3913
Denise Contos	PCI-SIM	(614) 424-3281
Dave Oiler	Sample Prep, Custodian	(614) 424-7962
Romona Mayer	QA/QC Advisor	(614) 424-7778

BUILDING DIAGRAM

Battelle-Columbus is a sensitive area with high security. The labs are located on three separate floors. The EI-MS and the PSI-SIM are located together in the same laboratory room. The NCI-SIM is located upstairs. The samples are in a freezer on another floor.

LAB TRACKING

As explained previously, the sample is logged in upon arrival, logged in and out of the freezer, logged into the project notebooks when

used, and logged into the instrument logbooks when analyzed. The sample history forms are used, and I reviewed them. Larry Slivon had some questions on the forms, whether all the information needed to be filled out. I will have to find out for him the information pertinent to BCD's role in the project.

BCD is considering a computerized form of tracking the sample along with the present method (paper trail). I feel the present method is adequate, but a second method could assist, and lower the chance of error.

ADDITIONAL COMMENTS

I was very satisfied with what I observed at Battelle-Columbus Laboratories. The arrangement and procedures were very excellent. Based on the areas evaluated, I saw no major areas of concern that warrant recommendations of change.

The only concerns I had did not involve BCD directly. Those were: 1.) the lack of a shipping list sent with the sample extracts sent from AWL. 2.) the lack of both collection reports and shipping reports involving the EPA spiked samples (resins and soils). These were given to Rocky Roades of the EPA at the December meeting. 3.) The preparative work on the resins needs additional attention. There was enough background interference in the resins that investigation into the preparation of the resins (clean-up before spiking or using for real samples) should have high priority.

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JAN 26 1989

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MEMORANDUM, 24 JANUARY 1989, FLOYD MCMULLIN, SUBJECT: REPORT OF
SITE VISIT TO SUNSET LABORATORIES 23 JAN 89 [sic]

ENCLOSURE 3

ENVIRONMENTAL LABS INC.
FIELD OFFICE
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JAN 23 1989

MEMORANDUM

TO: Environmental Labs. Inc.

ATTN: Dr. Gary Booth

FROM: Floyd McMullin

DATE: 24 January 1989

RE: Report of Site visit to Sunset Laboratories 23 Jan 89

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DATA MANAGEMENT CENTER

JAN 26 1989

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This report will be basically that information which is contained in my logbook. I will however make additions, changes and corrections as I feel necessary for clarity. This visit was conducted with Robert Cary.

LOI STATUS ON SITE: As of this time he has not written LOI. I discussed the basic need for these to be completed in the near future. We also discussed the differences between an SOP and an LOI in that an LOI may be altered based on changed needs and techniques being employed. We went over the instructions enclosed with the correspondence sent to him dated 22 Dec 88. He said he would complete them shortly and forward to either ELI or SNL or wherever as directed.

FIELD/LAB SAMPLING: Sample for analysis consists of a 1x1 1/2 cm piece of the quartz glass filter to be analyzed. The actual section of the filter used is selected at random and cut from the rest of the filter utilizing a cutting punch device.

FIELD/LAB ANALYSIS: After having manually placed the filter sample in the furnace and initiating the sequence on the control computer the rest of the analysis is fully automated under the computer's control. The basic sequence is that the sample is heated from room temperature through three different temperature levels (200, 400, and 700 degrees C approximately) while in an atmosphere of pure helium. During this time carbon compounds are monitored and the exact reflectivity (color) of the filter is monitored using a laser/photoelectric detector system. Sample is then cooled and oxygen is introduced. This results in burning off the carbon as carbon dioxide which is converted to methane utilizing a catalytic methanizer which is monitored/measured by a photoionization detector. The entire process allows a differentiation of carbon compounds versus elemental carbon in the sample. Output from the system is via a computer printer and consists of both numeric (for key values) and overall graphic representation.

INSTRUMENT/METHOD CALIBRATION: At least two methods to assure proper measurement and instrument calibration are utilized. The first method utilizes an automatic carbon insertion at the end of the analysis procedure for each sample. This value must read

LAB DIAGRAM: He will complete and forward to us.

RESEARCH JOURNAL: Utilizes a numbered page ledger to enter each sample run. Each days entries are noted and are in ink. The journal I examined had no pages missing so far as I was able to determine.

LAB TRACKING FORMS: Does not currently utilize and in-house form. He does log each sample into a register upon receipt. on the same register he also notes the day ran. and the date results are sent out. He currently utilizes the collection report received with each sample for assignment of the sample number. I discussed the need for some type of form showing receipt date, storage, removal, analysis, restorage date and signatures, and also the need for an area to document the final disposition of the sample. We went over the samples in the paperwork sent to him and he agreed to develop a form and utilize it.

MISCELLANEOUS: We discussed several miscellaneous items which need clarification for Mr. Cary.

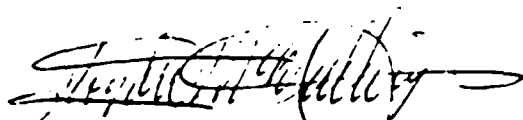
1. Where are reports to be sent? To Wayne Einfeld at SNL (since he's a subcontractor), or ELI, or to Don Johnson as stated when we were down at SNL in December? Who is to receive the floppy disk (discussed request for disk, he will supply in a Lotus ASCII file)?

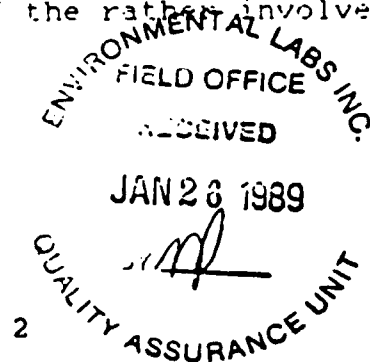
2. How long does he need to hold the original filter samples? He would prefer to not have to archive these due to the severely limited refrigerated storage space he has, and the large size of the filter storage bottles.

We need to advise Mr. Cary on these items as soon as possible.

OVERALL ASSESSMENT: Overall my assessment would have to be fairly good. Mr. Cary is personable and amenable to instituting changes in those areas where I noted deficiencies and discussed them with him. I do not feel that the lack of an equipment logbook is a problem of particular importance or concern in this setting. Mr Cary demonstrates a clear and ongoing concern for the accuracy of measurements done by his system.

I seriously doubt it will be feasible to do a systems audit of Sunset Laboratory. This is based on the fact that Mr. Cary is one of the few experts in the field and his custom equipment and software would require and extremely involved audit to discern the algorithyms being used to derive the final result, as well as a very good understanding of the rather involved hardware used.


Floyd W. McMullin Jr.
Quality Assurance Officer



ENVIRONMENTAL LABS, INC.
DATA MANAGEMENT CENTER

JAN 26 1989

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JAN 26 1989

REC. BY DLA

within a certain specified range or it indicates the unit is malfunctioning. The second method of calibration is to place a measured quantity of a carbon compound solution of a known concentration on a blank filter sample. This is then dried and subjected to analysis. As this sample contains a known quantity and concentration the amount of carbon may be predicted. If the sample fails to analyze within an acceptable error range ($\pm 5\%$) all sections of the analysis equipment are checked/adjusted/ repaired/replaced, etc. as needed.

PREVENTIVE/CORRECTIVE MAINTENANCE: Has ongoing systems monitoring. In the event of problems immediate repairs or maintenance is performed.

INTERNAL QC PROCEDURES: Utilizes automatic carbon spikes at completion of each sample as noted previously, and the spiked filters as noted previously. He mentioned that Wayne Einfeld had discussed and was supposed to be supplying a solution or solutions of carbon compound for blind analysis. He currently utilizes a sucrose solution because of the excellent pyrolysis characteristics of the material.

SAMPLE PREPARATION AND STORAGE: There is no preparation of the samples involved. All filters are initially prepared (cleaned) by AWL and they ship the used filters to him for analysis. The samples as received are stored in amber glass jars in a standard upright refrigerator.

PREPARATION AND USE OF SPIKED SAMPLES: See previous sections on calibration and QC procedures.

INSTRUMENT/EQUIPMENT SELECTION AND USE: Unit is a totally custom designed and built unit by R. Cary. Stated was developed over a number of years to its present state. Also made the comment that there was probably only one or two other comparable systems in the world.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION:
Detection limit is 0.2 mcg/cm²
Reproducibility is $\pm 5\%$

SAMPLE HANDLING AND TRANSPORTATION: Sample are handled only by Robert Cary (Signature, etc. is in the logbook). Currently no transport of shipments is done by Sunset Labs.

DATA REDUCTION AND ANALYSIS: Automated computer analysis and printout at the time of the sample run. System utilizes custom software designed and developed by R. Cary.

NOTES ON OTHER ITEMS:

LOGBOOKS: There is no logbook for this piece of apparatus. Mr. Cary does not keep one as it makes and prints a calibration check with each sample run.

MEMORANDUM, 24 JANUARY 1989, FLOYD MCMULLIN, SUBJECT: REPORT OF
SITE VISIT TO OREGON GRADUATE CENTER

ENCLOSURE 4

ENVIRONMENTAL
RECEIVED

JAN 26 1989

ENVIRONMENTAL
DATA MANAGEMENT

JAN 26 1989

QUALITY ASSURANCE UNIT
BY 10 MEMORANDUM
TO: Environmental Labs. Inc.

REC. BY DCA

ATTN: Dr. Gary Booth

FROM: Floyd McMullin

DATE: 24 January 1989

RE: Report of Site visit to Oregon Graduate Center

This report will be basically that information which is contained in my logbook. I will however make additions, changes and corrections as I feel necessary for clarity. This visit was conducted with Robert Daluge and Reinhold Rasmussen.

LOI STATUS ON SITE: No LOI are available at this time. Dr. Rasmussen has been busy dealing with schedule, sample number changes, construction of additional sampling equipment, etc. I did not pursue this subject based on previous discussions I had had with Dr. Rasmussen on the subject.

FIELD-LAB SAMPLING: Field sampling is accomplished by two different methods. One utilizes an evacuated cylinder (32 l & 6 tanks) which is opened at specified times during the test (generally background, inhomogeneous, and homogeneous). Upon opening of the cylinder by either manual or remote control the cylinder fills to ambient atmospheric pressure. The second method (800 ml SF₆) utilizes a transfer pump to fill the tanks to approximately 15 psig at specified times following the test detonation.

Lab sampling of the 32 liter tanks is accomplished by introducing a quantity of Baxter's HPLC solvent (Lot AS819 was used for the initial series of tests). Tank is then placed with the lower portion of the tank immediately above water heated 40-45 degrees C. while the upper portion of the tank has moist towels (cooling) applied to it. The unit is maintained thus for a period varying from a minimum of six hours to a period up to approximately 12 hours. This allows the solvent to evaporate and condense inside the tank. Upon completion of the tanks in the same test gang, all solvent is transferred to one tank and then to a sample bottle for shipment to AWL. This results in a small loss of solvent during the transfers. They are currently investigating utilizing a different method given to them by Dr. Broadbent. However, this as yet has not been tested as so specialized equipment must be constructed. The basis of the idea is that rather than introducing solvent into the cylinder in liquid state it would be introduced as a vapor only with the cylinder in an inverted (valve down) position.

As the person (Bob Watkins) who has done the sampling and analysis of the six liter tanks was not available I was not able to pursue the exact procedures employed on them.

JAN 26 1989

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The 800 ml of samples, since they are gaseous and pressurized are connected directly to a gas chromatograph for direct sampling and analysis.

Upon completion of refluxing or analysis all cylinders are cleaned and prepared for re-use.

FIELD/LAB ANALYSIS: All analyses are accomplished utilizing one or more gas chromatographs. The types currently in use at ECL are Perkin Elmer 3920, Carlo 211M, Hewlett Packard 5790A and a Shimadzu GC-mini 2. A reduction gas detector manufactured by Trace Analytical model RGD-2 is also used. Data from these devices is fed into a Hewlett Packard integrator for printout. The models currently in use are 3390A, 3396A, and 3388.

INSTRUMENT/METHOD CALIBRATION: In all systems repeated calibration/standardization sample runs utilizing a test gas of known concentration is done. Generally this is done after every fourth sample run. This allows checking on an ongoing basis to see if the calibration is drifting.

PREVENTIVE/CORRECTIVE MAINTENANCE: As all the devices in use are of solid state construction no routine preventive maintenance is required. In the event of equipment failure the problem is found, corrected, the instrument recalibrated, and placed back in service.

INTERNAL QC PROCEDURES: Other than the routine calibration checks during sample runs I was not made aware of any additional methods employed.

SAMPLE PREPARATION AND STORAGE: Preparation of the sample was covered under the lab sampling section. There is no preparation required on the 800 ml SF₆ samples. I am not aware of what, if any preparation is required on the six liter cylinders which are analyzed for volatiles. As mentioned previously, after analysis the tanks are prepared for re-use so there is not long term storage of the samples.

PREPARATION AND USE OF SPIKED SAMPLES: As noted previously concentrations of a known amount are routinely run. I was not made aware of any blind samples, although I recall that the EPA was to be preparing both 32 and 6 liter samples for recovery and analysis.

INSTRUMENT/EQUIPMENT SELECTION AND USE: Instrumentation in use is as noted in the Field/Lab Analysis section. It is outside my expertise to judge whether or not the actual operation and applications are correct.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION: I forgot to cover this area with the DGC personnel.

SAMPLE HANDLING AND TRANSPORTATION: Samples are handled by only four people (Signatures are in logbook). They are Dr. Reinhold

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Rasmussen, Robert Daluge, Don Stearns, and Bob Watkins. All transport of samples is done through either UPS Overnight or Federal Express.

DATA REDUCTION AND ANALYSIS For the type of analysis performed there is no apparent specialized analysis of results required. SFA concentrations are plotted graphically on a time line and analyzation for specific compounds results in either detection with a concentration or no detection.

NOTES ON OTHER ITEMS

LOGBOOKS: Logbooks for individual pieces of equipment are not kept. Calibration runs are kept with the analyzation runs done at the same time.

LAB DIAGRAM: Not obtained.

RESEARCH JOURNAL: These I have seen appeared to be intact, with all entries in ink.

LAB TRACKING FORMS: So far as I can determine no forms of any type are used to track the cylinders in-house. Prior to shipment out in the field all cylinders numbers are logged. When used in the field the data on each tank (number, date, time, etc.) is either recorded in the research journal, on the tank label, or both. Upon return to the lab they are processed (analyzed) as soon as practicable. The information used is that of the tank number and the information on the tank label. They also check to assure that all tanks sent out are received back at OGC. As noted previously, following analysis the tanks are cleaned for re-use. This effectively negates there being any "sample" to track, store, etcetera after completion on analysis.

MISCELLANEOUS: Dr. Rasmussen has several items of question/concern that need to be addressed and responded to.

1. Wants new sample bottle from AWL. He considered the initial shipment deficient for the fact that the caps were without exception either very loose or off all of the bottles received. Consequently he feels that they are most likely all contaminated with unknown materials from the pressure changes they underwent during air shipment. Additionally, some of the bottles shipped were received broken. He would like twelve additional bottles that have been cleaned, the caps placed on very snugly, masking tape seal around the base of each cap, and each bottle placed in an individual resealable plastic bag.

2. He also needs an address where the refluxed material from the 32 liter tanks is to be sent. UPS will not accept items for shipment unless it has an address they can physically locate. Box number is not acceptable.

3. With all the changes and additions they are not really certain

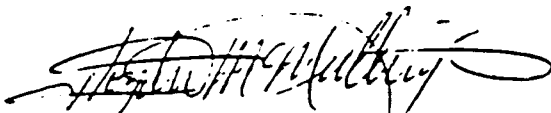
exactly what samples they're to be taking. and when. particularly for the rocket fuel burns. They don't have any idea how long the actual burn is expected to last.

Their general understanding is that they will take nine 32 liter tank samples during each of the detonations. These will all be taken at one time. during the homogeneous phase. They also understand that they are to take 27. 32 liter tank samples all at the same time during the final rocket fuel burn. Since the total number of tests has now been expanded far beyond the original design and test plan they have no documentation to go by.

OVERALL ASSESSMENT: Overall Dr. Rasmussen and Robert Daluge seem to be rather harried and frustrated with all the changes in schedule and what they are expected to do. They do state that they are doing their utmost to meet the changing requirements. but the time crunch for equipment preparation is their worst problem.

As I was unable to directly observe the majority of the procedures they use I cannot make definitive statements regarding their entire operation. That which I was able to observe (SF₆ analysis while down at SNL) appeared to be carefully and accurately done.

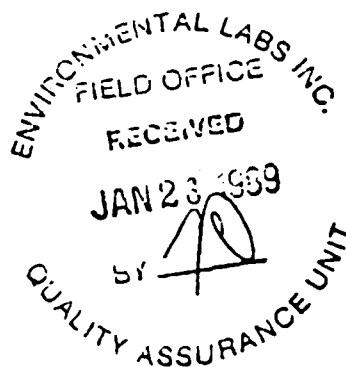
Due to the already serious tension level existing in trying to meet the requirements of the test I did not pursue nor point out with any emphasis those areas wherein OGC is deficient according to the EPA QA guidelines. I felt this would only serve to increase the frustrations and animosity which seems to be very near the surface with these people.


Floyd W. McMullin Jr.
Quality Assurance Officer

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MEMORANDUM, 24 JANUARY 1989, SUBJECT: MISCELLANEOUS ITEMS FROM OGC
SITE VISIT

ENCLOSURE 5

MEMORANDUM

TO: Environmental Labs. Inc.
ATTN: Dr. Gary Booth
FROM: Floyd McMullin
DATE: 24 January 1989
RE: Miscellaneous items from OGC site visit

I spent some time discussing the tank return needs for the various tests with Robert Daluge (he's leaving this Friday morning to go to SNL). He will need us to ship the 32 liter tanks back on overnight delivery following each of the first 3 detonations. This is needed to allow time for Dr. Rasmussen to reflux the tanks and get them back down to SNL for the final burn which will require 27 of the 32 liter tanks.

I also discussed the collection reports for the ganged 32 liter tanks. I told him we would use a single report for each group of tanks taken at the same time. This will both reduce paperwork, and also help avoid confusion, as ganged tanks are relaxed into a single sample.

We also talked about him keeping Todd apprised of the approximate schedule of what was going on back at OGC in the hopes of being able to put someone on site there while the actual refluxing of the 32 liter tanks and processing of the 6 liter tanks is going on. If we are able to work this out it may necessitate having someone else at SNL during part of the second week while I'm at OGC or having someone else go up to OGC to observe.



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MEMORANDUM, 25 JANUARY 1989, TODD PARRISH, SUBJECT: AWL SITE VISIT

ENCLOSURE 6

MEMORANDUM

TO: Dr. Gary Booth
FROM: Todd Parrish
SUBJECT: AWL Site Visit
DATE: 25 January 1989

The purpose of this report is to discuss the 25 JAN 1989 site visit of Alpine West Laboratory. On 18 JAN 1989, we contacted AWL and arranged a time for me to visit their facilities. This site visit was conducted according to the EPA's recommended guidelines, and Environmental Laboratory's visit worksheet. This report is arranged after that worksheet, and the notes that I recorded.

LOI STATUS

All the procedures involving the preparation and storage of samples are covered in the present LOI. The LOI also includes cleaning and extraction procedures. All LOI pertaining to the analysis are also included. As far as I can determine, there are no deviations from the LOI. The actual LOI are placed in both the cleaning/extraction laboratory, and the analytical laboratory for reference.

FIELD/LAB SAMPLING

All samples were collected on site in New Mexico, and I carried them back to AWL. Upon receipt, they were extracted with acetonitrile, as explained by the LOI. While at AWL they retain their ELI number. The laboratory also uses an altered sample history sheet (included). This includes information concerning the sampling, extraction, and analysis information. After extraction, half the sample extract is sent to BCD for analysis. The other half is hand carried from the Widtsoe Building to the Eyring Science Center for storage.

The samples are kept presently in a freezer in Dr. Milton Lee's office, ESC 106, BYU. There is no sign in/out sheet utilized there. The complete sample is located in the freezer, and no separate aliquot is used for analysis. Due to the fact that only two people will analyze the samples, there are no tracking forms used by the laboratory. The sample analysis form is updated when anything is done with the sample.

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FIELD/LABORATORY ANALYSIS

LOI include the procedures for the analysis. During my visit, no work had been accomplished on the actual samples. Only work on the detection limits and quantification limits had been done by AWL. The actual sample analysis will begin around 25 JAN 1989.

All data from the SFC/MS is stored on computer disks. When these are full, they are backed up on computer tape. At the present time, these are in Chris Rouse's desk.

INSTRUMENT CALIBRATION

This is performed daily by Chris Rouse. The standard that they use to calibrate the SFC-MS is FC 43. There is not an actual logbook, but there is a compilation of the daily calibration results. In the future, the laboratory personnel might want to sign and date these results (hard copies).

PREVENTIVE MAINTENANCE

Preventive maintenance is done regularly by trained technician. There is a logbook by the instrument that contains all work performed on the machine.

INTERNAL QUALITY CONTROL

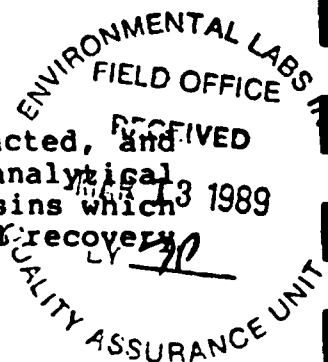
Chris Rouse, Karin Markedis, and Dr. Milton Lee will all determine what QC measures will be taken. Chris Rouse stated that standards will be analyzed every other day. Also, the MSD system and its results will be used in comparison with those of the SFC-MS.

SAMPLE PREPARATION AND STORAGE

The resin sample is extracted using a Soxhlet extractor, as explained in the LOI. The sample extraction, then is split into halves, one half going to BCD, and the other to Dr. Milton Lee's laboratory. They are stored there until analysis. The temperature of the freezer is taken once a month, but there is no record of it.

PREPARATION OF SPIKED SAMPLES

Resins will be spiked with appropriate standards, extracted, and analyzed to determine percentage recovery, and analytical efficiency. In addition the EPA will provide unknown resins which have been spiked with appropriate standards for analytical recovery.



data.

INSTRUMENT SELECTION

Alpine West Laboratory is utilizing a Finnagin Mass Spectrometer, and a Lee Scientific SFC. There is also a Hewlett Packard MSD.

DETERMINATION OF DETECTION LIMITS AND LIMITS OF QUANTIFICATION

At the time of my visit, the work on the detection limits and the limits of quantification had been completed, but there were not hard copies of all of the results. I saw two print-outs, and Chris Rouse informed me that they were presently in the 200 - 20 picogram/ml range of limit of detection. She also mentioned that this would improve as they started using the chemical ion detection method. The detection limits will be three times the background noise as the different ion method is utilized.

I did not have the opportunity to look at the quantification limits that had been finished as there was not a hard copy available to observe.

SAMPLE HANDLING AND TRANSPORTATION

The original samples were collected by Chris Rouse in Albuquerque. They were then brought back to AWL by van. The collection reports and shipping reports concerning AWL are all accounted for. The tank extracts from OGC arrived without ELI collection reports and shipping reports.

As mentioned before, all samples from the Bang Box are hand carried by Alpine West Laboratory. The samples sent to Sunset Laboratory, and BCD are sent Federal Express. I have overseen most of these shipments.

DATA REDUCTION AND ANALYSIS

This has not been done as no analysis has been preformed on the actual samples.

LOGBOOKS

I saw both Chris Rouse and Michael Dee's logbooks. Each page is a separate day, and all entries are done in black ink. As far as I can see, they are following LOI number 11.

As previously mentioned, there is not an actual notebook for the

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calibration of the SFC-MS. As a suggestion, I would recommend a notebook next to the instrument that records all the work that is performed on it.

PERSONNEL

Milton Lee	(801) 378-4338
Karin Markedis	(801) 378-2135
Chris Rouse	(801) 378-4466
Michael Dee	(801) 378-4466

OTHER

All entries in the notebooks, and logbooks are in black ink. The project notebooks are all intact, and appear to be up to date. As mentioned before, the sample analysis form is used instead of the tracking and sample history forms.

COMMENTS

I was satisfied with what I observed at Alpine West Laboratory. The set-up is excellent, and procedures proscribed in the LOI are in use. The LOI however, might need to be revised as they are now using dichloromethane as a solvent in both cleaning and extracting instead of acetonitrile.

Robert Rouse

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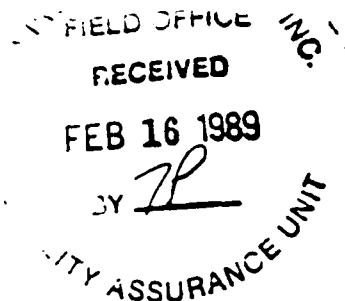
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MEMORANDUM, 31 JANUARY 1989, FLOYD W. MCMULLIN, SUBJECT: REPORT OF
SITE VISIT TO LAWRENCE BERKLEY LAB (LBL) [sic]

ENCLOSURE 7

MEMORANDUM



TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Floyd W. McMullin

RE: Report of site visit to Lawrence Berkley Lab (LBL)

DATE: 21 January 1989

The following is my report of the site visit to LBL on the 31st of January 1989. I met with Robert Glaugue and Linda Sindelar and spent several hours discussing various aspects of the project and checking the assorted areas of concern for quality assurance. For purposes of review and discussion I will basically duplicate the content of my logbook. The only changes that will be incorporated would be spelling and grammatical corrections, and if appropriate, more definitive explanations for clarity if I feel it is warranted.

The areas that I've written about basically correspond to the listing of components of a good quality assurance plan as listed in the EPA guidelines, and in the directions for preparation of a QA plan (Appendix to our QA plan).

LOI STATUS The LOI utilized for the studies is the standard procedure as spelled out in the X-Ray Fluorescence Analysis (XRFA) QC Procedures document as previously submitted. After having witnessed and reviewed the actual procedures with laboratory personnel the only variation from the documentation that I found (and not an important one) is that they are now utilizing a 20 MByte Bourne.11 box mass storage system rather than the 5 MByte removable hard disks as specified in the document. Copies of the procedure are kept in the lab, and are available for review if needed. As these people have been doing this type of analysis since 1972 their procedures are very standardized for all samples.

FIELD/LAB SAMPLING: As the test procedure employed is non-destructive to the filters being tested, no sampling is employed, rather the entire filter and holder are utilized.

FIELD/LAB ANALYSIS: A 5 cm² area of the filter is illuminated by secondary x-rays of various intensities. This is more fully covered in the previously mentioned QC procedures documentation. So far as I was able to determine, there is no variation from the procedure as described. In addition to the procedures outlined in the QC document Mr. Glaugue reviews the output data for each sample to see that the results are reasonable. If there are any questions

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from their prospective that may reanalyze a sample to verify findings.

INSTRUMENT/METHOD CALIBRATION: The calibration procedures utilized for this system are as specified in the QC procedures document and also in the published book: R.D. Glaugue, B.B. Garrett, and L.Y. Goda, in "X-Ray Fluorescence Analysis of Environmental Samples", T.G. Orpway, Ed. Ann Arbor Science, Ann Arbor, MI., 1977. A copy of the chapter pertaining to the calibration is attached to this report. I will not elaborate on the rather extensive procedures other than to say they appear well thought out, thorough, and very well done.

PREVENTIVE/COORECTIVE MAINTENANCE: The unit is monitored routinely for the quality of data output. If the output varies beyond the routine adjustment tolerances then specialized equipment repair personnel from LBL will check all phases of the unit to determine the cause and repair the unit.

INTERNAL QC PROCEDURES: Twice a day there are two separate quality checks run on the apparatus. The first consists of running the standard calibration three element filter (S, Cu, and Ag) for which absolute calibrations have been determined. Additionally a multi-element filter containing known quantities of 19 elements is run. Output data is checked against known values and the unit is adjusted if required.

SAMPLE PREPARATION AND STORAGE: Samples are stored in the containers as received. They are loaded into slide carrier trays for analysis and upon completion of analysis are returned to their original containers.

PREPARATION AND USE OF SPIKED SAMPLES: They do not prepare or use in-house spiked samples. Mr. Glaugue said he would be glad to run any provided to him, or re-submit a previously analyzed specimen under a different ID for re-analysis to see that their output is consistent. He also said that he would prefer that they not be aware of the spike or resubmitted samples. That way they wouldn't treat them any differently than other samples.

INSTRUMENT/EQUIPMENT SELECTION AND USE: The unit in use was designed and built at LBL by the instrument and techniques group. The unit currently in use has a high sensitivity when compared to other units in use/available.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION: The sensitivity limits for the 30 elements are all expressed as ng/cm².

Al=150	Si=40	S =15	Cl=12	K = 6
Ca= 5	Ti=30	V =20	Cr=15	Mn=12
Fe= 12	Ni= 6	Cu= 6	Zn= 6	Ga= 4
Ge= 3	As= 3	Se= 2	Br= 2	Rb= 2

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Sr= 3 Pb= 7 Zr= 8 Mo= 6 Ag= 5
 Cd= 6 Sn= 8 Sb= 8 I =12 Ba=35

The degree of accuracy varies somewhat depending upon the density of the element. Al= \pm 20% and Si= \pm 10%. All others fall within the range of \pm 1-3%.

SAMPLE HANDLING AND TRANSPORTATION: In the lab the samples are handled exclusively by Linda Sindelar. All samples are logged in, stored in a limited access lab, loaded into sample carrier trays, analyzed, transferred back into their original containers, and then shipped back to the sending lab.

DATA REDUCTION AND ANALYSIS: Is accomplished using custom software developed at LBL. The basic equations are covered in the QC procedure document in greater depth. The validity of the method is beyond the scope of this review.

This completes the evaluation based on the QA plan requirements. The material following covers various areas of our interest as specified in their headings.

LOGBOOKS: A large logbook/journal is utilized for all entries pertaining to the analyzation process. This includes sample number, date, repairs/adjustments to the unit, etc. There are also separate books of calibration runs, sample receipt logs, etc. The only variation noted is that all work I saw was done in pencil to allow for corrections of miscopied/written information. This appears to be and have been the practice since the beginning of the system (I checked a logbook from 1972). After having observed the amount of detailed information being recorded, and having witnessed the need to correct miscopied information, I can well understand the preference for pencil entries. Their logs when completed are neat and easy to read. If they were having to do these corrections, etc. in ink it would seriously degrade the readability of the logbook. I did not raise this issue with the LBL personnel as I did not feel this was essential as the information is also recorded in computer data and other logs as a double check and cross-reference.

PERSONNEL WORKING WITH SAMPLES: There are only two persons who have contact with the sample. They are Robert D. Giaque and Linda Sindelar (Signatures are in my logbook). Address is Lawrence Berkley Laboratory, Building 70 Room 275, Berkley, CA, 94720. Phone number is 415-486-5658.

BUILDING DIAGRAMS: This was not available at the time of my visit. Mr. Giaque will prepare one and forward it to use when complete.

RESEARCH JOURNALS: No individual journals are utilized. The large logbook mentioned previously serves this purpose.

LAB TRACKING: Individual tracking forms are not utilized.

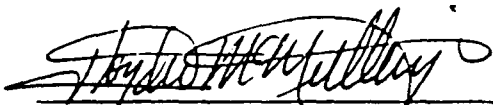
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beyond the initial log-in and assignment of a testing sequence number. Pertinent information is logged in several places during the entire sequence and cross referenced and checked by both computer (automatically) and Linca Sindelar to insure the accuracy of all information. Based on the exacting nature of the records keeping I observed and reviewed I would say that individual forms would not improve on their current system.

OVERALL ASSESSMENT/RECOMMENDATIONS: This laboratory is a well organized and run operation. They maintain very high standards of quality control and accuracy. Although they have a couple of minor departures from desired QA procedures (pencil in logbook and no individual sample tracking forms), I would recommend against requesting they change their system for our study. My basis of this is that their current system works very well, and requesting changes in their procedure (if they were willing to implement them) would tend to increase the chance of errors occurring due to the problems associated with altering a well established routine system. It is also my personal belief that they would most likely not be willing to institute appreciable changes as our samples constitute a very small percentage of the total samples they analyze.



Floyd W. McMullin Jr.
Quality Assurance Officer

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MEMORANDUM, 08 FEBRUARY 1989, FLOYD MCMULLIN, SUBJECT: ADDENDUM
TO SITE REPORT FOR OREGON GRADUATE CENTER

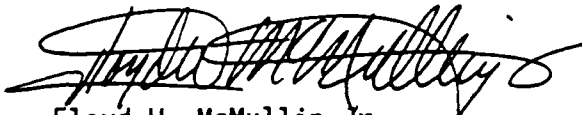
ENCLOSURE 8

MEMORANDUM

TO: Environmental Labs, Inc.
ATTN: Dr. Gary Booth
FROM: Floyd McMullin
DATE: 08 February 1989
RE: Addendum to site report for Oregon Graduate Center

As noted in my site report for the Oregon Graduate Center, I failed to get the information pertaining to Limits of Detection/Limits of Quantification while at the center.

I contacted Dr. Reinhold Rasmussen by phone the day following my site visit to obtain this information. He informed me that their Limits of Detection were 0.1 to 0.2 ppb with an accuracy of ± 15 to 20 percent. This range is based on the different materials they may be attempting to detect. He also stated the total explanation for the various tests and materials was rather complex and involved, but the above values basically covered the gist of it.

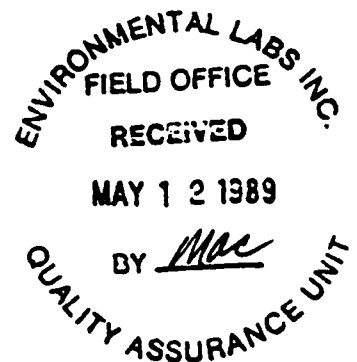

Floyd W. McMullin Jr.
Quality Assurance Officer

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LETTER MD-77B, FEBRUARY 15, 1989, U.S. ENVIRONMENTAL PROTECTION
AGENCY, ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT
LABORATORY, RESEARCH TRIANGLE PARK, NORTH CAROLINA, W/ENCLOSED
RESULTS OF OB/OD AUDIT AT SANDIA NATIONAL LABORATORIES

ENCLOSURE 9



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

February 15, 1989

Mr. Don Johnson
c/o AMFAC Hotel
2910 Yale Boulevard S.E.
Albuquerque, NM 87106

Dear Don:

Enclosed is a copy of our audit report. The results show how well the monitoring effort is being handled by you and your contractors. I would like to commend you on your organizational meetings each morning at 7. The number of contracts involved in this effort requires a good deal of coordination. Planning meetings each day give the contractors and yourself the opportunity to exchange ideas and to determine what is expected that day and in the near future.

I was impressed with the cooperation between contractors at the field site. The tight quarters in which they all had to operate could create personnel problems, but each knew his job and they coordinated with each other to ensure smooth project operation. For a laboratory that we have no contact with and is an independent air monitoring facility, Wayne Einfeld and his group at Sandia Labs were doing an excellent job in gathering the data in a timely fashion. The graphs Wayne presented show not only the data's quality and repeatability, but the speed with which it was processed.

I do feel that you should get some spares for equipment that might be more susceptible to vibrations encountered aboard the plane. Howard Crist will be sending you a report on the organics and the spiked samples.

If you have any questions, please feel free to call me at (919) 541-2205.

Sincerely,

A handwritten signature in cursive script that reads "Bill".

William F. Barnard
Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Enclosure

INTRODUCTION

During the period Feb.6-8, 1989 personnel from EPA's Quality Assurance Division at the Research Triangle Park, N.C. conducted a performance and systems audit of the OB/OD program at the Sandia National Laboratory's facility in Albuquerque, N.M.. The performance audit consisted of challenging each of the OB/OD continuous analyzers to known pollutant concentrations of SO₂, O₃, NO, and NO₂. The CO and CO₂ audits were conducted through the use of mailout disposable cylinders. The flows for each of the particulate and XAD cartridge samplers were also audited.

During the systems audit the Test Design Plan for the Preliminary Test Phase "A" was reviewed and checked against what actually occurred during one of the test blows. A number of documents were reviewed. These included the laboratory notebooks for the continuous monitors, the particulate samplers, and the volatile organic compounds, as well as QA/QC data and graphs of continuous monitoring data from earlier detonations.

SUMMARY and RECOMMENDATIONS

The audit results for the continuous gaseous monitors and the particulate sampler flows were excellent, as can be seen in Tables 1 and 2. The slopes of all the regressions of analyzer response on audit concentrations were within 7%. The flow differences were all less than 10%. Part of the error associated with the first two VOST samplers may be attributed to decrease in flow with time through the sampling media.

Sandia Labs had noticed such a phenomenon and it was apparent to the auditors as the audit progressed. VOST #2 was audited first, and its flows were higher than VOST #1. The same loaded XAD cartridge was used in line with both samplers to approximate the normal sampling train. A time lapse of 15-20 minutes occurred between the two audits. The other VOST samplers did not have sample media in line, only the mass flow meters, thus creating a different type of flow situation. It is also recommended that the 10 liter dry gas meter be calibrated in the 100l/min range, as the accuracy numbers for this meter were better at the lower flows. Generally a dry gas meter should only be used in the range between 3 times and at 1/3 of their face value.

The data for the three blows on Jan.31, Feb.2, and Feb.6, all show excellent repeatability. The Feb.6 data also shows how well Sandia Labs can retrieve and process the data that is under their direct control. A review of a number of laboratory notebooks indicated that good records were being kept for the continuous instrumentation and the particulate analyzers. Records of calibrations, zero and spans, and instrumental problems were documented.

It was noticed that there were relatively few pieces of spare equipment on hand. It is recommended that a spare VOST sampler be purchased and any other equipment which has been giving problems to the program since its inception. The instrumentation has had to cycle through temperature extremes and the auditors feel that the instrumentation may begin showing more serious problems when it starts sampling onboard the aircraft.

The 80 liter bag had been removed and the instrumentation had been shifted around to accommodate the changes. It was not obvious to the audit team that these changes had been documented. It is recommended that all changes in the program be documented and a reason given for each change.

NO-NO2-NOX AUDIT RESULTS

Table 1

Site Name Date	Model S/N	Range ppb	Audit Conc. ppb	Analyzer Response ppb	Difference ppb	Difference percent	Regression-Response on Audit Conc. Slope Intercept Corr. Coeff.
SANDIA	CSI 1800	0-5000	NO				
2/7/89			1020	1043	23	2.3	0.950 72.0 0.9999
			820	855	35	4.3	
			620	657	37	6.0	
			430	475	45	10.0	
			0	76.2	76.2	-	
			NO2				
			420	398	-30	-7.0	0.934 -1.63 0.9999
			281	262	-19	-6.8	
			0	-2	-2	-	
			NOx				
			1020	1051	31	3.0	0.952 85.5 0.9998
			820	872	52	6.3	
			620	683	63	10.0	
			430	485	55	12.7	
			0	87	87	-	

SO2-O3 AUDIT RESULTS

Table 1 (con't)

Site Name Date	Model S/N	Range ppb	Audit Conc. ppb	Analyzer Response ppb	Difference ppb	Difference percent	Regression-Response on Audit Conc. Slope Intercept Corr. Coeff.
SANDIA	TECO	0-500	SO2				
2/7/89	43		422	422	-	-	1.003 -0.82 0.9999
			203	202.5	-0.5	-0.25	
			103	103.8	0.8	0.78	
			61	61.9	0.9	1.5	
			0	-3.0	-3.0	-	
SANDIA	TECO 100	0-1000	O3				
2/6/89			199.8	190.6	-9.0	-4.5	0.942 1.23 0.9997
			99.9	94.6	-5.4	-5.4	
			70.2	65.6	-4.6	-6.5	
			30.0	28.0	-2.0	-6.6	
			15.3	16.0	0.7	4.5	
			0.0	3.8	3.8	-	

OB/OD FLOW AUDIT RESULTS

Table 2

Sampler I.D. Date	Sampler Flow l/min	Audit Flow l/min	Percent Difference
Vost #1	107.0*	98.8*	8.3
Vost #2	110.8*	103.3*	7.3
Vost #3	62.9	67.2	-6.4
Vost #4	65.0	71.6	-9.2
Bubbler Pump #7	1.94*	1.99*	-2.5
Bubbler Pump #9	1.81*	1.85*	-2.2
Bag Teflon XRF Sampler	51.9	52.8	-1.7
Bag SEM Sampler	15.5	15.3	1.3
Bang Box SEM Sampler	2.88*	2.87*	0.4

* Flows compared at ambient conditions

All others compared at 25deg C. and 760mmHg

LETTER MD-77B, MARCH 2, 1989, U.S. ENVIRONMENTAL PROTECTION AGENCY.
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY.
RESEARCH TRIANGLE PARK, NORTH CAROLINA, W/ENCLOSED REPORT OF
SPIKED SAMPLES

ENCLOSURE 10



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

March 2, 1989

Mr. MacDonald Johnson
Commander Headquarters AMCCOM
AMSMC-DSM-D
Rock Island, IL 61299

Dear Mr. Johnson:

The results of the contractor laboratories analyses of the spiked samples for the OB/OD project are summarized in the enclosed report.

If you have any questions or comments on the data, please let me know (919) 541-2723.

Sincerely yours,

Howard Crist

Howard Crist
Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Enclosure

cc: Dr. H. Smith Broadbent
Dr. Rai Rasmussen
Dr. Laurence Slivon
Dr. Milton Lee

Results of Analyses of Audit Samples for Open Burning/Open Detonation Project

In December, 1988 a number of spiked audit samples were provided to the contractor analytical laboratories for the Open Burning/Open Detonation (OB/OD) project. Selected media (soil and resins), 6 l canisters and 32 l tanks were spiked with several OB/OD target compounds. A sample of the solution used to spike the soils, resins and 32 l tank was also provided. The 6 l canisters were prepared from an NIST-traceable gas cylinder.

Spiked resins analyses results were submitted by Battelle (Columbus Division) but not by Alpine West Laboratories¹. Analytical data of the spike solution and 32 l tank extracts were submitted by both laboratories. Soil analytical results were not reported by either laboratory. The Oregon Graduate Center reported data for the 6 l canisters.

Discussion of Results

Canisters

Table 1 shows the results of the 6 l canister analyses and the percent differences between the spiked and reported concentrations. The two canisters contained duplicate concentrations of several volatile organic compounds (VOCs). All compounds in the audit mixture were correctly identified.² The results for the more volatile compounds of the mixture are very good, but the less volatile compounds exhibit a pattern of decreased recovery. Most of the data is negatively biased. Repeatability of analyses for the more volatile compounds is also very satisfactory. Previous experience with analysis of VOCs indicates that the less volatile compounds have a greater tendency to absorb on the surface of equipment during analysis (transfer lines, etc.). Because the data for the highly volatile compounds are significantly better it appears that a similar phenomenon may have contributed to the poorer results of a few of the components of the audit mixture.

Spike Solution

The results on the analyses of the spike solution are listed in Table 2. Phenol, 2,4-dinitrophenol and dibenzo (a,h) anthracene was not reported by Alpine West Laboratories and Battelle did not report 2,4-dinitrophenol. Diphenylamine was not in the spike solution, but 15 µg/ml was reported by Alpine West. Battelle's overall results were satisfactory except for the high value reported for 4-nitrophenol. Alpine West's data was significantly, negatively biased for three of the four compounds they reported.

¹They reported that interferences prevent an accurate analysis.

²1,3-butadiene was correctly reported in the samples, but no reliable reference values can be assigned due to its instability.

32 1 Tanks

The two 32 1 tanks were spiked with identical compounds and concentrations. They were supposed to be analyzed separately, but the individual extracts of the tanks were combined before analysis. The results appear in Table 3. No values for phenol and 2,4-dinitrophenol were submitted by Alpine West. Battelle did not report the presence of 2,4-dinitrophenol in the tank extracts. The data was predominantly, negatively biased, indicating incomplete extraction of compounds from the tanks.

Spiked Resins

Tables 4 and 5 list the data for the spiked resins analyses. The presence of 2,4-dinitrophenol was not reported. N-nitroso-diphenylamine was not quantified in one sample and benzo (a) pyrene was not analyzed in two samples because of reported interferences. Significant levels of phenol and 4-nitrophenol were reported on the blank resin. The results for phenol and 4-nitrophenol had excessive positive biases which probably resulted, at least in part, from the relatively high levels of these compounds found on the blanks. Most of the other values were reasonably close to the spiked amounts.

The results for the resins analyses indicate that the spiked compounds were more efficiently extracted from Porapak than from XAD-2 (Table 5). Based on these data, a review of the resin extraction procedure should be conducted in an effort to improve the extraction efficiency for XAD-2. Perhaps a more effective solvent could be used.

Table 1
Analyses of VOC Audit Canisters (Oregon Graduate Center)

	<u>Spiked, ppb</u>	<u>Reported, ppb</u>		<u>Difference, %</u>	
	CQ 142 CQ 338	CQ 142	CQ 338	CQ 142	CQ 338
vinyl chloride	3.4	3.7	3.6	8.8	5.9
bromomethane	3.6	3.8	3.6	5.6	0.0
trichlorofluoromethane	3.8	3.7	3.6	-2.6	-5.3
carbon tetrachloride	3.5	2.8	3.0	-20	-14
methylene chloride	3.5	3.4	3.1	-2.9	-11
chloroform	3.6	4.7	4.5	30	25
1,1,1-trichloroethane	3.6	3.4	3.2	-5.6	-11
1,2-dichloroethane	3.7	2.7	2.4	-27	-35
benzene	3.3	2.5	2.2	-24	-33
toluene	3.7	2.6	2.1	-30	-43
1,2-dibromoethane	3.9	2.4	1.9	-38	-51
tetrachloroethylene	3.8	3.3	3.0	-13	-21
chlorobenzene	3.8	2.6	2.0	-32	-47
o-xylene	3.7	1.8	1.0	-51	-73
trichloroethylene	4.2	3.5	3.3	-17	-21
1,2-dichloropropane	3.8	2.8	2.3	-26	-39
ethylbenzene	3.6	1.9	1.2	-47	-67

Table 2
Analyses of Spike Solution

	Actual Conc. ug/ml	Reported ug/ml		Difference, %	
		Alpine West	Battelle	Alpine West	Battelle
phenol	29	NR	20	-	-31
4-nitrophenol	22	1.5	50	-93	127
* 2,4-dinitrophenol	19	NR	NR	-	-
N-nitroso-diphenylamine	22	1.9	26	-91	18
benzo (a) anthracene	23	17	23	-26	0.0
benzo (a) pyrene	27	13	26	-52	-3.7
dibenzo (a,h) anthracene	20	NR	16	-	-20
diphenylamine	a	15	-	-	-

a - not spiked

* not a target analyte

NR - not reported

Table 3
Analyses of Two 32 l Tank Spikes

	Spiked, ug (duplicates)	Reported, ug		Difference, %	
		Alpine West	Battelle	Alpine West	Battelle
phenol	29	NR	14	-	-52
4-nitrophenol	22	3.2	10	-85	-55
2,4-dinitrophenol	19	NR	NR	-	-
N-nitrosodiphenylamine	22	4.4	19	-80	-14
benzo (a) anthracene	23	24	21	4.3	-8.7
benzo (a) pyrene	27	16	23	-41	-15
dibenzo (a,h) anthracene	20	8	9.4	-60	-53
diphenylamine	a	5.5	-	-	-

a - not spiked

NR - not reported

Table 4
Spiked Resins Analyses

	Spiked, μg		Reported, μg (Battelle)			
	2PB	1PB	2PB	3XB	1PB	2XB
	3XB	2XB				
phenol	7.2	36	17	44	36	120
4-nitrophenol	5.5	28	61	25	290	200
2,4-dinitrophenol	4.6	23	NR	NR	NR	NR
N-nitroso-diphenylamine	5.6	28	NQ	3.4	27	19
benzo (a) anthracene	5.8	29	6.1	3.6	30	16
benzo (a) pyrene	6.8	34	6.4	NQ	34	NQ
dibenzo (a,h) anthracene	5.0	25	10	3.2	31	18

Blanks	μg	
	1XB	3PB
2-nitronaphthalene	1	-
phenol	9.5	18
4-nitrophenol	-	4.5

NR - not reported

NQ - not quantified-interference

Table 5
Spiked Resins Analyses

	Difference, %			
	2PB	3XB	1PB	2XB
phenol	136	511	70 ^{5.0}	233
4-nitrophenol	1000	354	935	614
2,4-dinitrophenol	-	-	-	-
N-nitroso-diphenylamine	-	-39	-3.6	-32
benzo (a) anthracene	5.2	-38	3.4	-45
benzo (a) pyrene	-5.9	-	0.0	-
dibenzo (a,h) anthracene	-	-36	24	-26

Note: No correction for blank background was made.

PB - Porapak

XB - XAD-2

LETTER MD-77B, MARCH 7, 1989, U.S. ENVIRONMENTAL PROTECTION AGENCY,
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY,
RESEARCH TRIANGLE PARK, NORTH CAROLINA, W/ENCLOSED REPORT OF CO
AND CO₂ AUDITS

ENCLOSURE 11



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

March 7, 1989

Mr. Dennis Morrison
Sandia National Laboratories
Division 6321
P.O. Box 5800
Albuquerque, NM 87185

Dear Dennis:

Enclosed are the results of the EPA CO and CO₂ performance audit of the OBOD "Bang Box" monitoring project. The results were excellent. If there are any questions concerning the audit, please feel free to call me at 919-541-2205.

Sincerely,

A handwritten signature in cursive script, appearing to read "Bill".

William F. Barnard
Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Enclosure

cc: William J. Mitchell
W. Einfeld

Sandia National Laboratories OBOD Audit

Cylinder No.	Analyzer Conc. ppm CO ₂	Audit Conc. ppm CO ₂	ppm	Differences percent	Analyzer Conc. ppm CO	Audit Conc. ppm CO	ppm	Difference percent
3023	365	350	15	4.3	38.0	36.5	1.5	4.1
3012	363	350	13	3.8	37.9	36.5	1.4	3.8
1138	345	350	-5	-1.4	6.2	6.2	0.0	0.0
1001	348	350	-2	-0.6	6.2	6.2	0.0	0.0

MEMORANDUM, 12 MAY 1989, DR. GARY BOOTH, SUBJECT: INTERIM REPORT ON
SITE VISITS

ENCLOSURE 12

MEMORANDUM

TO: Don Johnson
FROM: Dr. Gary Booth
DATE: 12 May 1989
RE: Interim Report on Site Visits

The following is a report of the site visits conducted during the first phase (bang-box) of the OB/OD testing. During December and January, all six laboratories were visited by either Floyd McMullin or Todd Parrish. The laboratories visited were Sandia National Lab (SNL), Oregon Graduate Center (OGC), Battelle-Columbus Division (BCD), Lawrence Berkley Lab (LBL), Alpine West Lab (AWL), and Sunset Lab (SSL). An additional visit during the month of May was conducted at AWL and SSL.

This report will summarize the findings of the site visits according to the Good Quality Assurance Plan (EPA), and the project QA plan.

LOI STATUS

At the time of the initial site visits, all laboratories except OGC and SSL had submitted LOI. Since the initial site visit we have received the LOI from OGC. The LOI from SSL was obtained at the second site visit (9 May 89) and is undergoing technical assessment. Revisions to LOI appear to have been done as necessary to accommodate procedural changes incorporated in analyses as the project has progressed.

FIELD/LAB SAMPLING

This was covered in all the laboratories visited. Overall the methods of sampling appear to be as described in LOI.

FIELD/LAB ANALYSIS

This was also checked in all of the laboratories visited. Again the analysis methods/techniques are as described in the LOI with the various changes which have been incorporated.

INSTRUMENT CALIBRATION

All laboratories calibrate their instruments according to either the manufacturers specifications, or their own internal standards in the case of custom apparatus.

PREVENTIVE/CORRECTIVE MAINTENANCE

Corrective maintenance is performed on the instruments when needed. All labs regularly perform preventive maintenance on their instruments.

INTERNAL QC PROCEDURES

SNL, AWL, BCD, and LBL all have a person responsible for the QC procedure in the laboratory. OGC and SSL are using calibration checks and spikes respectively as QC procedures.

PREPARATION AND USE OF SPIKED SAMPLES

All laboratories except LBL utilize spiked samples that are prepared in the laboratory. LBL stated they would be glad to run any spikes provided to him. This same statement holds true for SSL. AWL, BCD, and OGC also run tests of spiked specimens received from EPA. I will however, note that the EPA failed to provide documentation with the samples they have furnished even though appropriate collection reports and shipping lists were supplied to their representative.

SAMPLE HANDLING AND TRANSPORTATION

In-house handling and custody was covered with each laboratory manager. Overall this was found to be adequate. Transportation of specimens between facilities is accomplished utilizing either Federal Express overnight delivery, or by courier.

DATA REDUCTION

Most of the labs stated that the methodology utilized for data reduction would be covered in the final LOI. OGC stated no specialized analysis was necessary for their results. LBL uses custom software, the equations are covered in their procedure document. SSL also utilizes a computer analysis with custom software which would be somewhat of a proprietary nature.

LOGBOOKS

All of the laboratories utilize logbooks of various varieties. LBL makes their log book entries in pencil which is covered in the site visit report for that facility.

DETECTION LIMITS/LIMITS OF QUANTIFICATION

AWL and BCD are still determining what their detection limits and limits of quantification are, and will be included in their final reports.

SSL states their detection limit is 0.3 ug/cm^2 with a precision of $\pm 5\%$, and an accuracy of $\pm 5\%$.

OGC stated it varies somewhat depending upon the material being looked for but the limit of detection range was 0.1 to 0.2 ppb with an accuracy of ± 15 to 20 percent.

LBL stated the detection limits for the 30 elements that they detect vary from 2 to 150 ng/cm^2 . The actual limit for each element will be included in the final report. The degree of accuracy for all elements except Al and Sn will be within the range of ± 1 -3%. Al = $\pm 20\%$ and Sn = $\pm 10\%$

SNL informed us that these would be covered in their final LOI.

MEMORANDUM, 12 MAY 1989, FLOYD MCMULLIN, SUBJECT: SUNSET
LABORATORY SITE VISIT 9 MAY 1989

ENCLOSURE 13

MEMORANDUM

TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Floyd McMullin

DATE: 12 May 1989

RE: Sunset Laboratory site visit 9 May 1989

ENVIRONMENTAL LABS, INC.
DATA MANAGEMENT CENTER
MAY 12 1989
REC. BY *Mac*

ENVIRONMENTAL LABS, INC.
FIELD OFFICE
RECEIVED
MAY 12 1989
BY *Mac*
QUALITY ASSURANCE UNIT

I met with Robert Cary of Sunset Lab to go over OB/OD testing to this point so far as his lab is concerned. At this point all samples (filters) submitted to him from the initial Bang-Box trials have been completed, and data from his analysis has been sent to Wayne Einfeld of SNL (A copy of his report has been received by ELI for archival purposes).

As relates to his data I was requested to obtain verification as to some of the abbreviations and notations on his data sheet. They are OC = Organic Carbon, EC = Elemental Carbon, INST.BLNK refers to an instrument blank which is a clean quartz filter ran as an internal zero check of his instrumentation. The notations of SUGAR plus an extension refer to the sucrose solution utilized as an internal quality control analysis. The extension refers to the concentration of sucrose in micrograms (i.e. 10UL=10mcg/ml). The use of sucrose as a reference check has been covered in my previous site visit report, and in the LOI for this lab.

I received the LOI for SSL. Upon initial review it appears to be a complete document. It is attached for technical assessment and placement in the document files.

We discussed several items of interest as pertains to quality control of this project which will need to be addressed/corrected during any future testing.

1. Mr. Cary encountered glass filters instead of quartz fiber filters in part of the samples. These were ELI numbers 183, 184, 186, 189, 297, and 378. Upon checking records I found that these were all VOST sampling filters, and were all from the second series of test conducted during February 1989. I will need to follow up on this problem with AWL on my next visit. The main problem with the glass filters is that they melt during the testing process resulting in damage to the testing system. So far as I know SSL is the only lab that would be effected by this problem as their testing process results in heating the filter above it's melting point.

2. He asked me if the aluminum foil used to wrap the filters in during the second series of trials had been baked to remove any impurities. Having seen the methods employed by AWL on the test site I informed him it had not. He mentioned that this

should be considered in the future as he has tested aluminum foil and found that it has carbon material left on the surface as a residue from manufacturing.

3. Mr. Cary also mentioned that he had returned some of the filters to AWL at the request of Dr. Broadbent. He had also returned the collection reports with the samples. He said he was told that AWL had sent him the entire filter in error, rather than just a portion of it. This will need to be followed up with AWL as having had filters returned to them has resulted in no filter samples being retained for archival purposes for those filters that were returned. Additionally Mr. Cary did not have record of which filters were returned. This will need to be checked with AWL also.

Overall my assessment of Sunset Lab remains very good. So far as I can determine they maintain high quality and accuracy standards. I have no recommendations for changes at this facility.



Floyd W. McMullin Jr.
Quality Assurance Officer

ENVIRONMENTAL LABS, INC.
DATA MANAGEMENT CENTER

MAY 12 1989

REC. BY



ENVIRONMENTAL LABS, INC.
FIELD OFFICE
RECEIVED
MAY 12 1989
BY 
QUALITY ASSURANCE UNIT

APPENDIX E. CONSOLIDATED ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AEHA	U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, Maryland
AFB	Air Force Base
AMC	U.S. Army Materiel Command, Alexandria, Virginia
AMCCOM	U.S. Army Armament, Munitions and Chemical Command, Rock Island, Illinois
amino-PAH	aminopolycyclic aromatic hydrocarbons
ANOVA	analysis of variance
AP	ammonium perchlorate
APS	aerodynamic particle sizer
ASASP	active scattering aerosol spectrometer probe
AWL	Alpine West Laboratories, Provo, Utah
BB	BangBox
BCD	Battelle Columbus Division, Columbus, Ohio
BD	target analyte not found in concentrations above detection limits
BYU	Brigham Young University, Provo, Utah
CAA	Clean Air Act
CDD	chlorinated dibenzodioxin
CDF	chlorinated dibenzofuran
CI-SIM	chemical ionization, selective-ion monitoring
CSI	Columbia Scientific Instruments
C·V	concentration times cloud volume method
CWA	Clean Water Act
DMC	Data Management Center
DMPS	differential mobility particle sizer
DoD	Department of Defense
DPG	U.S. Army Dugway Proving Ground, Dugway, Utah
EC	electron capture <i>or</i> elemental carbon
ECD	electron capture detector
EDAX	energy-dispersive X-ray analysis
EER	Energy and Environmental Research Corporation, Irvine, California

EF	emission factor(s)
EI	electron impact
EI-MS	mass spectrometer used in the electron impact ionization mode
EI/MS	electron impact ionization/ mass spectrometry
EIS	environmental impact statement
ELI	Environmental Labs, Incorporated, Provo, Utah
EOD	explosive ordnance disposal
EPA	U.S. Environmental Protection Agency
EPO	Environmental Protection Office, U.S. Army Dugway Proving Ground, Dugway, Utah
ER	expansion ratio
FID	flame ionization detector
FSSP	forward scattering spectrometer probe
FTIR	Fourier Transform Infrared Spectrometry
FWAC	fixed-wing aircraft
GC	gas chromatograph(y)
GC-ECD	gas chromatography with an electron capture detector
GC-FID	gas chromatography with a flame ionization detector
GC/MS	gas chromatography-mass spectrometry
GLP	good laboratory practices
HE	high explosive
HMX	octamethylenehexanitramine
HNBB	hexanitrobibenzyl
HRGC/HRMS	combined capillary column gas chromatography/high resolution mass spectrometry
HS	high-speed
LASD	Los Angeles Sheriff Department
LBL	Lawrence Berkeley Laboratory, Berkeley, California
LC	liquid chromatography
LOD	limit of detection
LOI	letter(s) of instruction
NO _x	nitrogen oxide (s)

MR	multiple range
MRI	Midwest Research Institute, Kansas City, Kansas
MS	mass spectrometry (or mass spectrometer)
MSA	Mine Safety and Appliance Company
NA	not targeted for analysis <i>or</i> not applicable
NASA	National Aeronautical and Space Administration
NATICH	National Air Toxics Information Clearinghouse
NBS-SRM	National Bureau of Standards (now NIST)- Standard Reference Material
ND	no data <i>or</i> detection limit not determined
NEPA	National Environmental Policy Act
NF	not found in the sample matrix <i>or</i> not determined
NIST	National Institute of Science and Technology
nitro-PAH	nitropolycyclic aromatic hydrocarbons
NIOSH	National Institute for Occupational Safety and Health
NOSIH	Naval Ordnance Station, Indian Head, Maryland
NO _x	nitrogen oxides
NS	not sampled
OB	open burning
OB/OD	open burning/open detonation
OC	organic carbon
OD	open detonation
OGC	Oregon Graduate Center, Beaverton, Oregon
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbons
PANH	polycyclic aromatic nitrogen heterocycles
PAOH	polycyclic aromatic oxygen heterocycles
PCDD	polychlorinated dibenzodioxins
PCDF	polychlorinated dibenzofurans
PETN	pentaerythritol tetranitrate
PEP	propellants, explosives, and pyrotechnics
PIC	products of incomplete combustion
PICI/SIM	Positive ion chemical ionization/selective ion monitoring

PID	photoionization detector
PIP	product improvement program
PM	program manager
PMS	Particle Measuring Systems, Inc.
PUF	polyurethane foam
QA	quality assurance
QA/QC	quality assurance/quality control
QC	quality control
QAA	quality assurance agency
QAPP	quality assurance project plan
QAU	quality assurance unit
RCRA	Resource Conservation and Recovery Act
RDX	hexamethylenetrinitramine
RFD	Reno (Nevada) Fire Department
RIC	relative ion count
RSD	relative standard deviation
RTP	Research Triangle Park, North Carolina
SDPDA	Special Defense Property Disposal Account
SEM	scanning electron microscope/microscopy
SFC	supercritical fluid chromatography
SFC/MS	supercritical fluid chromatography/mass spectrometry
SF ₆	sulfur hexafluoride
SIM	selected-ion monitoring (or selective-ion monitoring)
SNL	Sandia National Laboratories, Albuquerque, New Mexico
SOP	standing operating procedures
SS	stainless steel
SSC	stainless steel canister
SSL	Sunset Laboratory, Forest Grove, Oregon
STEL	short-term exposure limit
STP	standard temperature and pressure (25°C and 760 torr)
TCD	thermal conductivity detector
TDP	test design plan

TEAD	U.S. Army Tooele Army Depot, Tooele, Utah
TECO	Thermo Electron Instruments (Company)
TECOM	U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland
THC	total hydrocarbon
TLV	threshold limit values
TNT	2,4,6-trinitrotoluene
TSC	technical steering committee
TSP	total suspended particulate
TWA	time-weighted average
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland
UV	ultraviolet
VOC	volatile organic compounds
VOST	semivolatile organic sampling train
VSDM	Volume Source Diffusion Model
XRF	X-ray fluorescence <i>or</i> X-ray fluorescence spectrometer

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APPENDIX F - DISTRIBUTION

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